Quantum Design

Magnetic Property Measurement System

SQUID VSM User's Manual

Part Number 1500-100, C0

Quantum Design

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Trademarks

U.S. Patents

5,053,834	High Symmetry DC Squid System
5,110,034	Superconducting Bonds for Thin Film Devices
5,139,192	Superconducting Bonds for Thin Film Devices
5,311,125	Magnetic Property Characterization System Employing a Single Sensing Coil Arrangement to Measure AC Susceptibility and DC Moment of a Sample (patent licensed from Lakeshore)
5,319,307	Geometrically and Electrically Balanced DC Squid System Having a Pair of Intersecting Slits

Application number 11/326,903 (filed Jan. 6, 2006) Superconducting Quick Switch.

Foreign Patents

Canada	2,089,181	High Symmetry DC Squid System
Japan	2,533,428	High Symmetry DC Squid System

C O N T E N T S

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Contents and Conventions

P.1 Overview

In this chapter we describe the scope of the manual, the conventions used and most importantly the safety guidelines. The SQUID VSM using cryogens and high power components, we strongly recommend to be aware of all hazards in order to prevent injuries and system damage.

P.2 Scope of the Manual

This manual discusses the MPMS SQUID VSM. It contains information about the basic functionality, describes the hardware that is unique to the system, and explains how to use the system and the SQUID VSM MultiVu software.

P.3 Conventions in the Manual

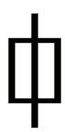
File menu	Bold text identifies the names of menus, dialogs, options, buttons, and panels used in the SQUID VSM MultiVu software.
File > **Open**	The > symbol indicates that you select multiple, nested software options.
`.dat`	The Courier font indicates file and directory names and computer code.
Important	Text is set off in this manner to signal essential information that is directly related to the completion of a task.
Note	Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances.

This symbol signals specific caution or conditions that could result in system damage, bodily harm, or loss of life.

This symbols signals electrical hazards that could result in bodily harm, or loss of life. Used at all accessible 200-230 V power outlets.

This symbol signals **cryogenic hazards** that could result in bodily harm and loss of life. Used wherever accessible parts could reach temperatures below 0°C (32°F).

This symbol signals **hot surface hazards** that could result in bodily harm and loss of life. Used wherever accessible parts could reach temperatures above 60°C (140°F).



This symbol signals information on **fusing**.

P.4 Safety Guidelines and Regulatory Information

Before using this product, please read the entire content of this User's Manual and observe all instructions, warnings and cautions. These are provided to help you understand how to safely and properly use the SQUID VSM and reach its best performances.

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse or improper operation of the system. Please contact your Quantum Design representative for any service issues.

This product is NOT operator-serviceable except for simple operations described in Appendix A.

Observe the following safety guidelines when you use your system:

- In case of emergency, switch the power off at the rear of the cabinet or unplug the main power cord from the laboratory power outlet.

- To prevent electrical shock, unplug the system before you install it, adjust it, or service it.

- The instrument must be plugged into a 200-230VAC 50/60HZ single phase appliance outlet fused to 16A (Outlet type IEC 60309, 200-250V, 16A, 2P+E, 6H, blue). See Appendix D.2.

- To prevent damage, always power down module bay tower when removing/installing modules.

- For continued protection against fire hazard, electric shock and irreversible system damage, replace fuses only with same type and rating of fuses for selected line voltage. Information about user-accessible fuses and their replacement is summarized in Appendix A.3.9.

- Direct contact with cryogenic liquids, materials recently removed from cryogenic liquids, or exposure to the boil-off gas can freeze skin or eyes almost instantly, causing serious injuries similar to frostbite or burns. Wear protective gear, including clothing, insulated gloves, and eye protection, when you handle cryogenic liquids.

- Transfer cryogenic liquids only in areas that have adequate ventilation and a supply of fresh air. Nitrogen and Helium gas can displace the oxygen in a confined space or room, resulting in asphyxiation, dizziness, unconsciousness, or death.

- Do not obstruct the cryogen exhaust lines. The exhaust should be visible to the user when operating the system.

- Keep this system away from radiators and heat sources. Provide adequate ventilation to allow for cooling around the cabinet and pump console. The distance between the system and wall should be at least 30 cm. (12 inches) in each direction. Do not obstruct the ventilation openings on the top of the cabinet.

- Do not obstruct the ventilation outlet located on the left side of the pump console and air intake at the rear. The clearance around the pump console should be at least 20 cm. (8 inches) in each direction.

- Do not obstruct or pinch the pump exhaust line located at the rear of the pump console.

- Refer to the manuals for the pump, monitor and printer for additional safety warnings and notices.

DECLARATION OF CONFORMITY

According to EU - Directives:
EMC: 89/336/EEC and 2004/108/EC & LVD : 73/23 and 93/68

Manufacturer

Name: Quantum Design Inc.
Address: Corporate Headquarters
6325 Lusk Boulevard
San Diego, CA 92121-3733
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Product Name

Model/Type: MPMS SQUID VSM, MPMS SQUID VSM EVERCOOL™
Brand Name: Quantum Design

Standards to which Conformity is declared

LVD
EN 61010-1 2nd ed. : 2001,
IEC 61010-1: 2001 (Second Edition)

EMC
EN 61326-1 :2006 Class A
EN 55011: 2007,
EN 55022: 2006,
IEC 61000-4-2: 1995/A1: 1998/A2: 2000,
IEC 61000-4-3: 2006,
IEC 61000-4-4: 2004 + Corrigendum 1: 2006,
IEC 61000-4-5: 2005
FCC Part 15B, AS/NZS CISPR 11

Documentation Reference

Test Reports No.
Nemko CB Test report No 79763 with amendment No 108152
Nemko EMC report No 2007 012109 Rev1 (MPMS SQUID VSM)
Nemko EMC report No 2008 07108152 (EVERCOOL™ PUMP CONSOLE)

I, the undersigned, hereby declare that the above defined product is conform to the above specified standards

Jerry D. Davis

Jerry Daviess, President

SAN DIEGO, CA; USA 11/13/08

Place and Date

C-CE-DOC-SVSM Rev. 02 October 27, 2008

C H A P T E R 1

Getting Started

1.1 Overview

In this chapter we give a brief system introduction and describe how to get started and to perform the most common tasks with your SQUID VSM. The instructions are meant to be brief in order to get a quick overview of the routine operations and to get familiarized with the system. More details are provided in the rest of the manual.

1.2 Introduction

The Quantum Design Magnetic Property Measurement System (MPMS) is a family of analytical instruments configured to study the magnetic properties of small experimental samples over a broad range of temperatures and magnetic fields. Automated control and data collection are provided by a computer and various electronic controllers. Extremely sensitive magnetic measurements are performed with superconducting pickup coils and a Superconducting Quantum Interference Device (SQUID). For this reason, the MPMS family of instruments are called SQUID magnetometers.

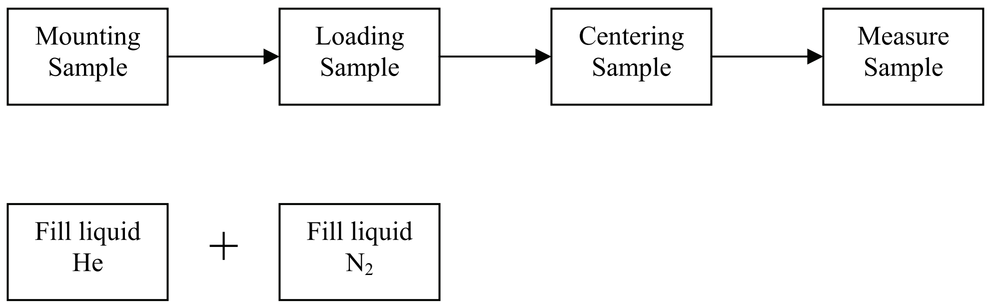
To optimize speed and sensitivity, the MPMS SQUID VSM utilizes some analytic techniques employed by Vibrating Sample Magnetometers (VSMs). Specifically, the sample is vibrated at a known frequency and phase-sensitive detection is employed for rapid data collection and spurious signal rejection. Unlike traditional (non-superconducting) VSMs, the size of the signal produced by a sample is not dependent on the frequency of vibration, but only on the magnetic moment of the sample, the vibration amplitude, and the design of the SQUID detection circuit.

The MPMS SQUID VSM utilizes a superconducting magnet (a solenoid of superconducting wire) to subject samples to magnetic fields up to 7 Tesla (70 kOe). The SQUID and magnet must both be cooled with liquid helium. Liquid helium is also used to cool the sample chamber, providing temperature control of samples from 400 down to 1.8 Kelvin. To help conserve liquid helium, the system is designed to use less costly liquid nitrogen to intercept heat bound for the helium tank. The SQUID VSM will only operate properly with both cryogens in use: liquid helium and liquid nitrogen.

For details on hardware and measurement methods we refer to chapter 3 and 4 respectively.

1.3 Common Tasks

The most common tasks necessary to perform a measurement are summarized in the following flowchart:



1.3.1 Mounting Samples

- For the most accurate magnetic moment measurements, measure the magnetic signature of the sample holder before mounting the sample and subtract its signal from the total signal.
- Mount samples very securely to a sample holder to achieve accurate magnetic moment readings. The sample holder will undergo vibration at 14 Hz. (GE) 7031 varnish is recommended, or a glue that will withstand the temperature extremes and sample holder thermal contraction during the experiment. 7031 varnish can be dissolved with alcohol or toluene to remove samples and re-use sample holders. Do not soak the entire sample holder in solvents like acetone—they may affect the adapter at the end.
- Mount samples on-axis with the sample rod to minimize dependence of the magnetic moment readings on the angular orientation (about a vertical axis) of the sample rod.
- Quartz and brass sample holders are supplied. The quartz holders are brittle and fragile, but have the smaller magnetic signature. For samples having a large moment, the brass sample holders will have negligible magnetic background and may be easier to handle.
- Use the supplied sample mounting station to place the sample at the proper location on the sample holder as shown below. The location markings are on a mirror to help eliminate parallax. Load the sample holder by retracting the spring-loaded end with your fingers and inserting the top end of the sample holder.

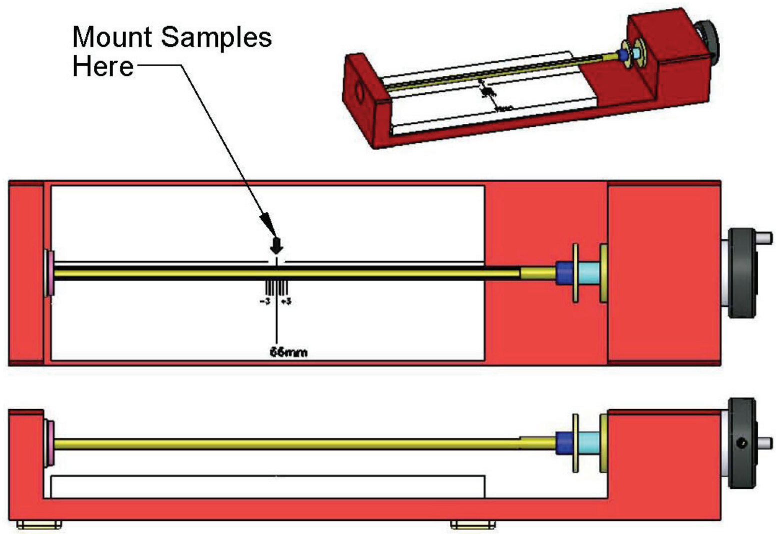


Figure 1-1. Sample mounting station.

- Custom sample holders may also be used, but accuracy and reproducibility of magnetic moment readings may be compromised. The supplied brass and quartz holders are manufactured to ensure optimal geometry, and the SQUID VSM software can accurately predict their thermal contraction in order to keep samples properly centered in the detection coils. User-designed sample holders may not meet these aims. For example, plastic drinking straws may be used, but are not recommended because:
    - It is difficult to mount samples rigidly enough to the straw to withstand the acceleration of the VSM oscillation.
    - It is difficult to secure straws to sample rods perfectly co-axially.
    - Plastic straws do not withstand the upper end of the instrument temperature range and will melt.
    - Above room temperature, the thermal expansion of plastic drinking straws is not predictable, and the auto-tracking algorithms used in the SQUID VSM to help track sample position do not apply.

1.3.2 Loading and Unloading Samples

1. Attach a new sample holder with mounted sample to a sample rod. Make sure that the mating surfaces of the adapters are clean and that they are screwed all the way in. When screwing your sample holder to the sample rod, hold the rod at the blue bearing.

Figure 1-2. Sample holder mounting.

2. Select the software menu item "Sample > Install/Remove…"

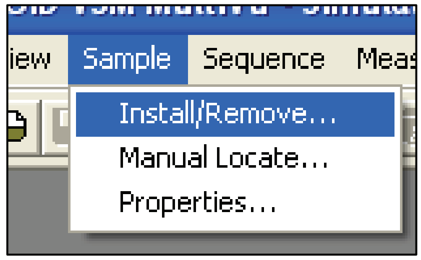


Figure 1-3. Sample installation menu.

The Install/Remove Sample Wizard begins. It guides you through the rest of the sample loading process, including warming the chamber to room temperature and venting the chamber, specifying a data file for saving measurement data, entering sample and sample holder information, and centering the sample. If you only wish to unload a sample, the wizard instructs you how to do this.

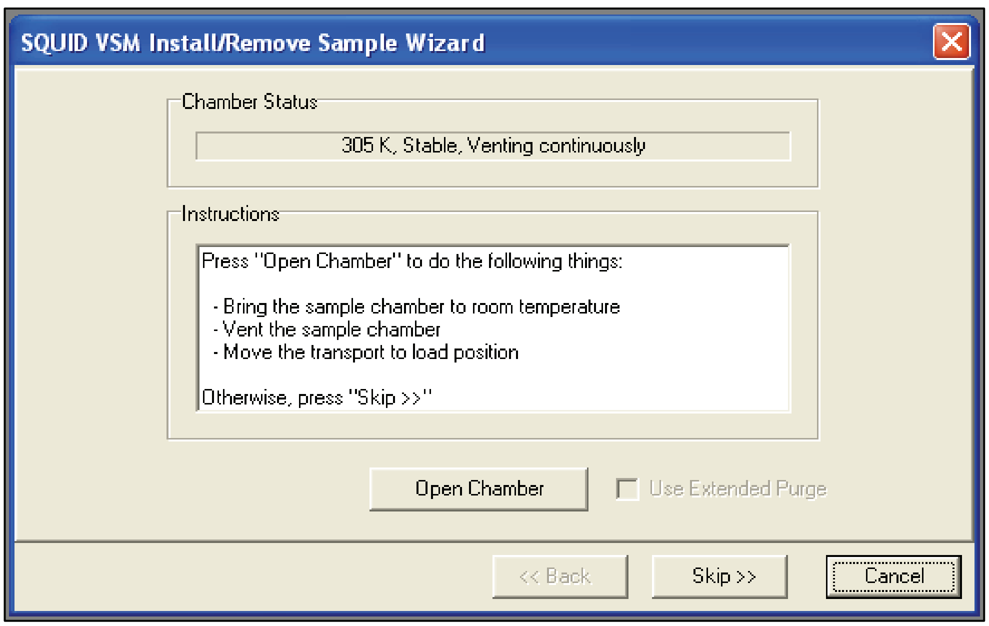


Figure 1-4. Sample installation wizard.

3. Follow the instructions in the wizard.

Alternatively, you may unload samples with the procedure below, or load samples without specifying data file, sample, or sample holder information. This procedure will not help you center your sample, either.

1. Stop any measurement in progress. Make sure the chamber temperature is above 295 K and below 315 K. Set temperature and wait if necessary, to prevent condensing or freezing air and moisture in the chamber.
2. Press "eject" 

The chamber vents continuously with helium and the VSM head moves to the top position.

3. Remove the cap from the VSM head.

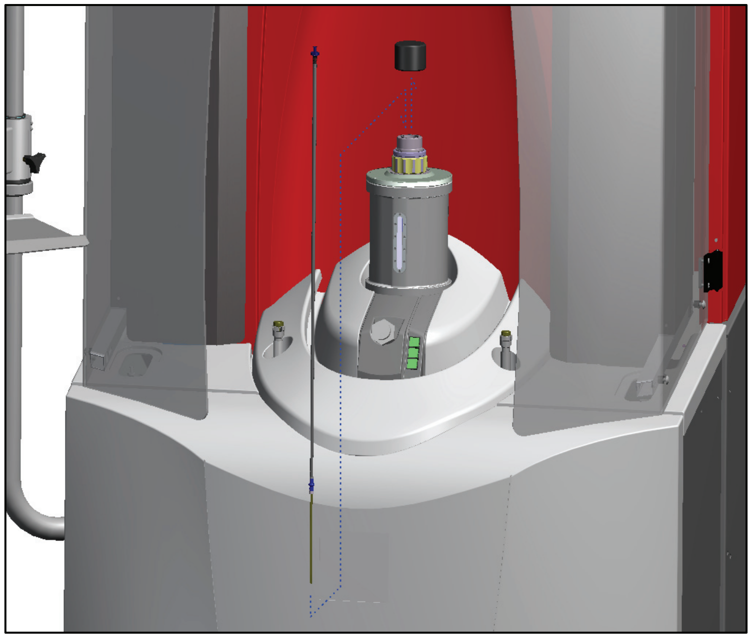


Figure 1-5. Sample rod installation.

4. Remove the installed sample rod. Insert a new sample rod into the chamber if desired.

5. Replace the cap and press "load"

   The chamber is purged with helium. You will need to specify a data file and center your new sample in order to perform measurements.

Never press "load" without having replaced the cap. Air will be sucked into the system and cause ice accumulation in the sample space.

1.4 Centering Samples

- The sample must be centered within the detection coils for accurate magnetic measurements. This can only be done if the sample is mounted near the correct location on the sample holder, as shown in the section "Mounting Samples" above.
- Whenever you load a new sample, specify the sample offset so that it may be properly centered during measurements. This is the distance from the bottom end of the sample holder to the (magnetic) center of the sample.

- The Install/Remove Sample Wizard helps you specify the sample offset. If you are not using the wizard, select "Sample > Manual Locate."
- The sample offset is defined as the distance from the bottom end of the sample holder to the sample location and is usually about 66 cm. The motor armature position should be roughly centered in the window of the VSM head.

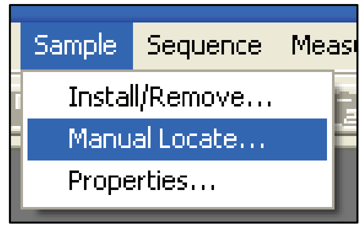


Figure 1-6. Manual locate menu.

- There are two different ways to enter the sample offset:
    - The most accurate technique is to **scan the sample**. The software moves the sample through the detection coils and analyzes the coil response to locate the sample. To perform a good centering scan you may need to induce a magnetic moment > 1×10^{-7} emu in your sample by applying a magnetic field, or by changing the temperature.
    - If your sample has a very weak magnetic response and inducing a magnetic moment is undesirable or impossible, scanning the sample may not work. If your sample does not have a dipole-like response, scanning the sample may not work. In these cases you can measure the sample offset with a rule or caliper before loading the sample and **enter the sample offset manually**.

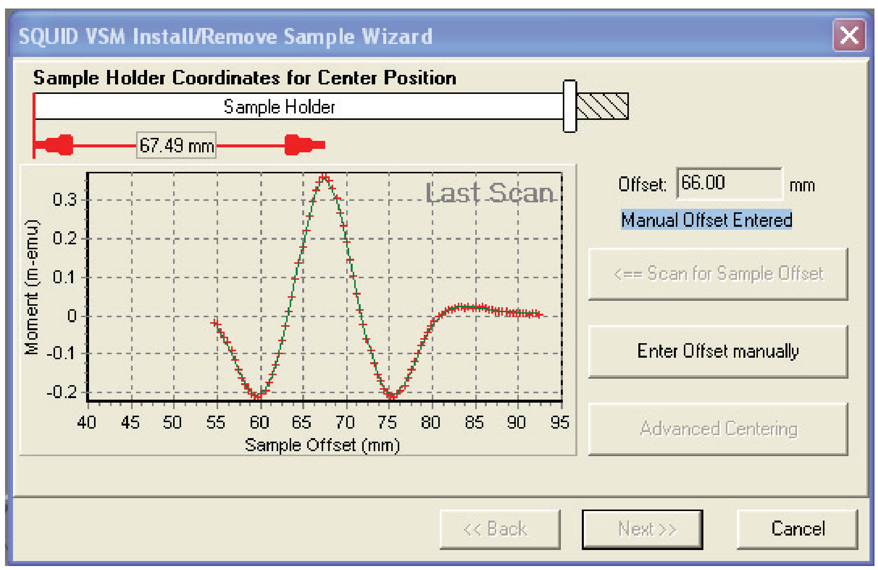


Figure 1-7. Sample centering scan.

1.4.1 Scan the Sample

1. Click "Scan for Sample Offset." The instrument moves the sample through the detection coils. It may scan the sample several times to achieve optimum signal.

2. The signal is plotted in red as a function of position, along with a dipole response fit in green. A good scan has one central peak with symmetric peaks on either side. The center peak is approximately 170% the magnitude of the outer peaks, and opposite in polarity. This is due to the geometry of the detection coils.
3. The instrument calculates the sample offset based on the fit to the data and asks you for confirmation to use the calculated value. Click "OK" only if the fit and calculated sample offset look correct.

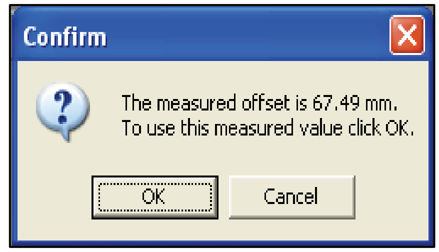


Figure 1-8. Sample offset confirmation.

1.4.2 Enter Sample Offset Manually

1. Set the temperature to 305 K and wait for the chamber to reach 305 K. The sample offset you measure at room temperature is not valid at other temperatures, due to thermal contraction of the sample rod. If you are using the Install/Remove Sample Wizard, it will set the temperature for you.
2. Click "Enter Offset Manually."
3. Enter the sample offset you measured. This is usually about 66 mm. For accurate measurements, it is important to enter the offset as close as possible to the true value.
4. Click "OK."

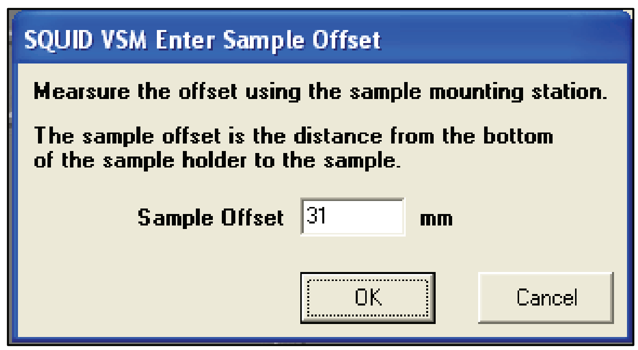


Figure 1-9. Manual sample offset.

1.4.3 Automatic Centering

- As the temperature of the sample changes, the dimensions of the sample rod and sample holder will also change, moving the sample away from the center of the detection coils. To compensate for this, use **automatic centering** to keep your sample centered during experiments.
- There are two automatic centering modes:
  - **Auto-Tracking** adjusts the sample location automatically to compensate for the thermal contraction of the sample rod and sample holder. The brass or quartz sample holder must be specified in the "Sample Properties" dialog.
  - **Auto-Scan** will periodically scan the sample at designated intervals to locate it. You may specify both time and temperature intervals. No magnetic moment data will be collected while each scan is being performed.

- ○ You may also **disable** automatic centering. This will reduce the moment accuracy and result in slight measurement errors if the chamber temperature changes.

- Specify the automatic centering mode each time you start a measurement or each time you place a measurement command or a center sample command into a sequence. The automatic centering setting is found on a "centering" tab for each measurement, or in a "centering" section on the "advanced" tab for some measurements.

1.4.4 Measuring Samples

1.4.4.1 IMMEDIATE MEASUREMENTS

- Initiate sample measurement with the menu item "Measure > Measure…" After you click the "Start" button, the measurement begins and the sample's temperature, magnetic field, magnetic moment, and moment standard error data are recorded in the data file you have specified.

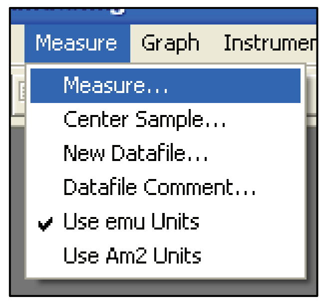


Figure 1-10. Measure menu.

- Specify "Continuous Measuring" for a continuous stream of data, or "Single Measurement" to collect a single data point.

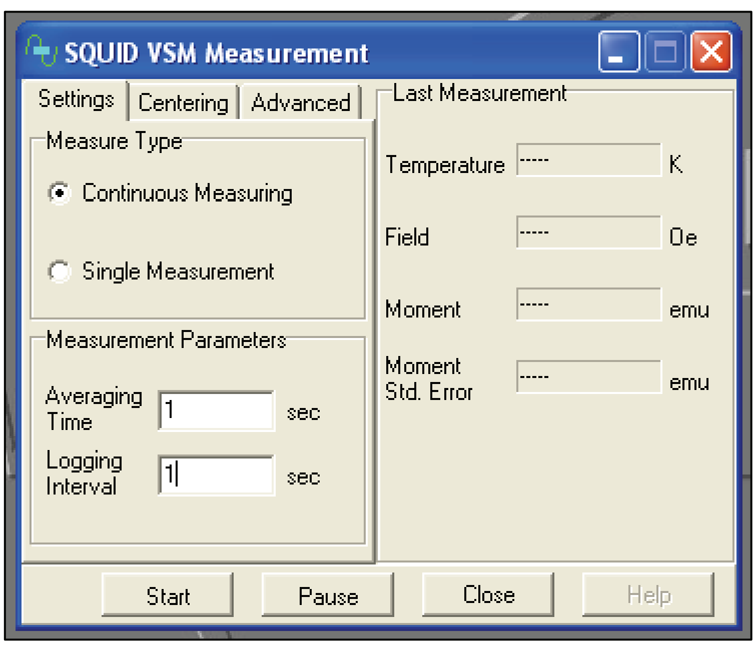


Figure 1-11. Measurement parameters.

- Specify the averaging time and the logging interval (for continuous measurement stream).
- On the “Centering” tab, specify which automatic centering method is used to keep the sample centered.
- On the “Advanced” tab, you may also set the vibration amplitude and SQUID range settings. 2 mm is the default vibration amplitude. The instrument achieves highest sensitivity with vibration amplitudes greater than 2 mm. (See chapter 4 for vibration amplitude considerations.)
- Set the instrument temperature or magnetic field to vary those parameters as desired.

1.4.4.2 SEQUENCE (AUTOMATED) MEASUREMENTS

- To automate the temperature and magnetic field variation and the data collection, use a sequence. (See chapter 2 for more about sequences.)
- The sequence commands “Measurement Commands > VSM > Moment vs. Field” and “Measurement Commands > VSM > Moment vs. Temp.” help automate very common magnetic measurements.

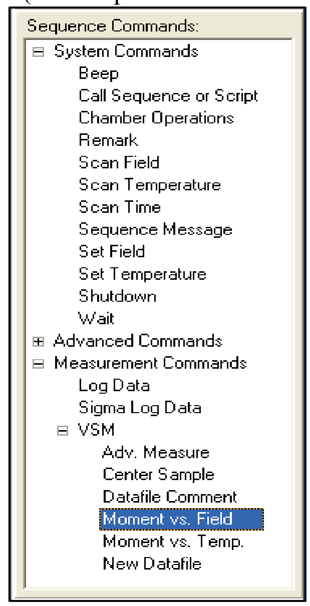


Figure 1-12. Sequence commands menu.

- For **Moment vs. Field** measurements, specify the shape and size of the magnetic field ramp by clicking and dragging in the “Select Start/End Quadrant” area to highlight the desired ramp shape, and by entering the H_{max}, H_0, and H_{min} fields. Enter the field sweep rate and choose whether to stabilize at each measurement field or continue sweeping the field during measurements (sensitivity will be reduced.) Select the data spacing, averaging time, number of fields between H_{min} and H_{max} OR the field increment, and the number of measurements to repeat at each field.
- The areas on the right side of the **Moment vs. Field** window indicate the approximate fields where measurements will be performed and the approximate time to perform the series of measurements, based on all of the settings.

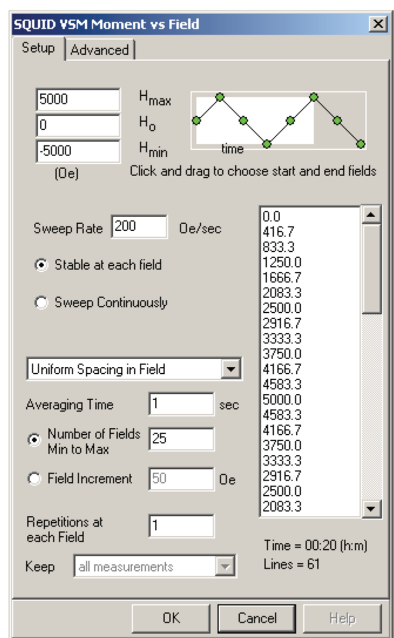


Figure 1-13. M(H) measurement setup.

- For **Moment vs. Temperature** measurements, specify the starting and ending temperature, temperature sweep rate, and whether to stabilize at each measurement temperature or continue sweeping the temperature during measurements. Select the data spacing, averaging time, number of temperatures at which to measure OR the temperature increment, and the number of measurements to repeat at each temperature.

- The areas on the right side of the **Moment vs. Temperature** window indicate the approximate temperatures where measurements will be performed and the approximate time to perform the series of measurements, based on all the settings.

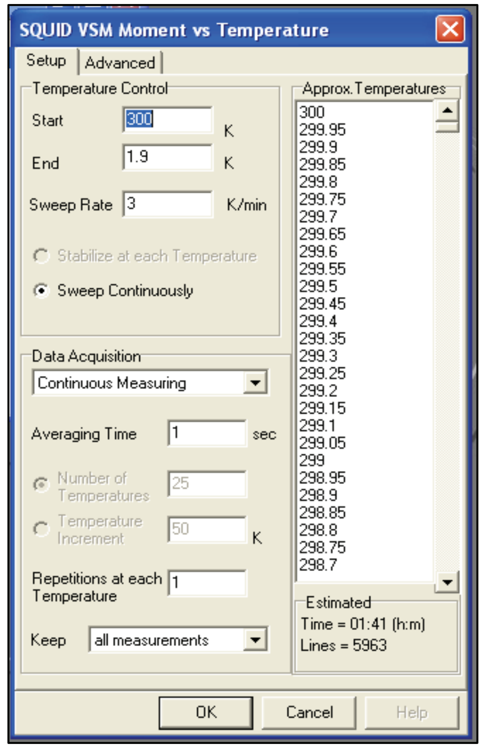


Figure 1-14. M(T) measurement setup.

1.5 Filling the Helium and Nitrogen Tanks

W A R N I N G !

Cryogens such as liquid helium and liquid nitrogen pose the following dangers: (1) They can cause serious burns due to extreme cold. (2) They may expand explosively in closed containers if warmed above their boiling point. (3) Their vapors may displace the breathable air in a closed room. (4) Liquid or vapors may backstream out of fill ports when removing caps/plugs/transferlines.

To guard against these dangers, wear eye protection and protective clothing such as gloves and covered shoes when working with these cryogens. Also ensure that the room has good ventilation. Never disable relief valves, burst disks, or other safety devices on cryogen vessels and transfer equipment.

Experienced personnel should perform or supervise these activities.

When transferring helium, the probe top plate is heated to avoid moisture and ice collection close to electronic equipment. Beware, surfaces of the top plate might reach high temperatures.

The MPMS SQUID VSM usually requires nitrogen about as often as it requires helium. It is however recommended to transfer the nitrogen and helium on separate days. Since there is no nitrogen level meter and the presence of nitrogen is critical for low temperature operation and minimizing consumption of helium, the nitrogen should be added once a week. Do not use a high pressure (~200 psi, ~14 bar) liquid nitrogen storage dewar, but instead stay with the lower pressure (22 psi, 1.5 bar) rated nitrogen containers. The helium should be added as needed.

If you fill the tanks while the instrument is measuring, measurement accuracy and precision may be compromised.

Several indicators help you to track your helium level:

a. **Status bar:** The MultiVu status bar shows the remaining helium volume in liters (see section 2.2.5).

b. **Color bar:** The color bar in the status bar indicates the urgency for helium refill.

   Green: Helium level sufficient for proper operation.

   Yellow: System is still operational but helium should be filled soon.

   Red: Helium needs to be filled immediately. The system will shutdown and return to operational only once helium is filled up.

c. **Status light:** The lower status light on the probe follows the same scheme as the color bar in the status bar (see section 1.6).

1.5.1 Fill the Liquid Nitrogen Tank

Open the fill port on the top of the nitrogen tank to determine if there is liquid nitrogen in the tank. If there is liquid nitrogen in the tank there will be a slight overpressure and gas will be forced out when you open it. If the tank has no liquid nitrogen there will be no overpressure or there will be a vacuum created by the liquid helium evaporation which chills the nitrogen tank below the nitrogen boiling point—air will be sucked in when you open the tank. This vacuum effect can also be generated immediately after transferring liquid helium into the helium tank because the high flow of cold helium gas during the transfer may also cool the nitrogen tank below the nitrogen boiling point. Therefore, this check is only reliable two hours or more after transferring liquid helium into the instrument. (It is best to perform this check before starting to fill the helium tank.)

1. Connect the hose to the nitrogen storage dewar.

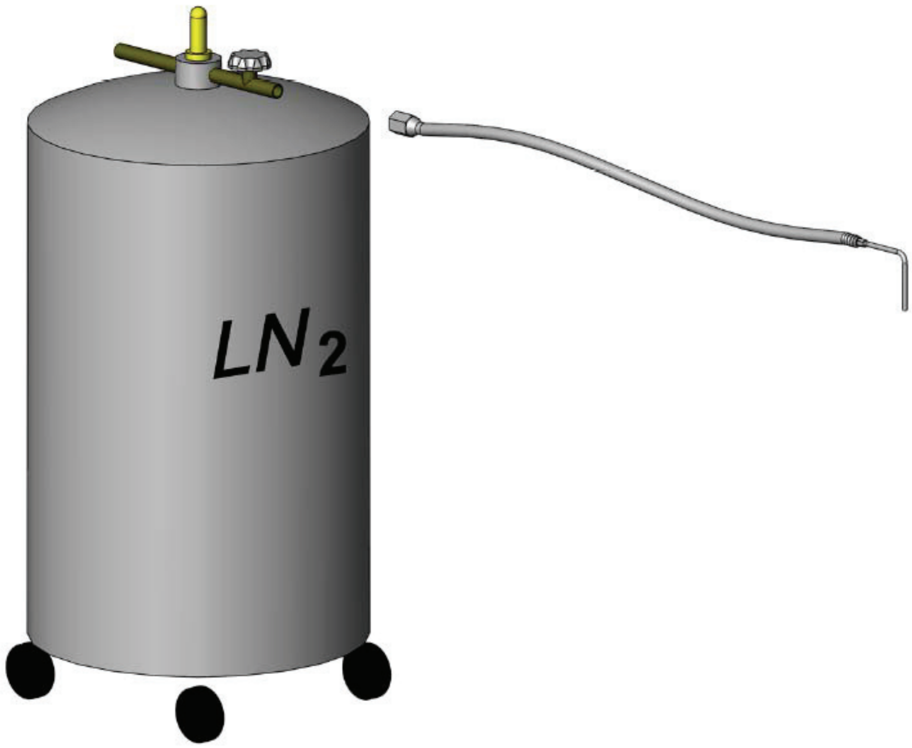


Figure 1-15. Liquid/Nitrogen transfer tank. (only use low pressure containers p< 22psi, 1.5 bar)

2. Open one nitrogen fill port and insert the hose.

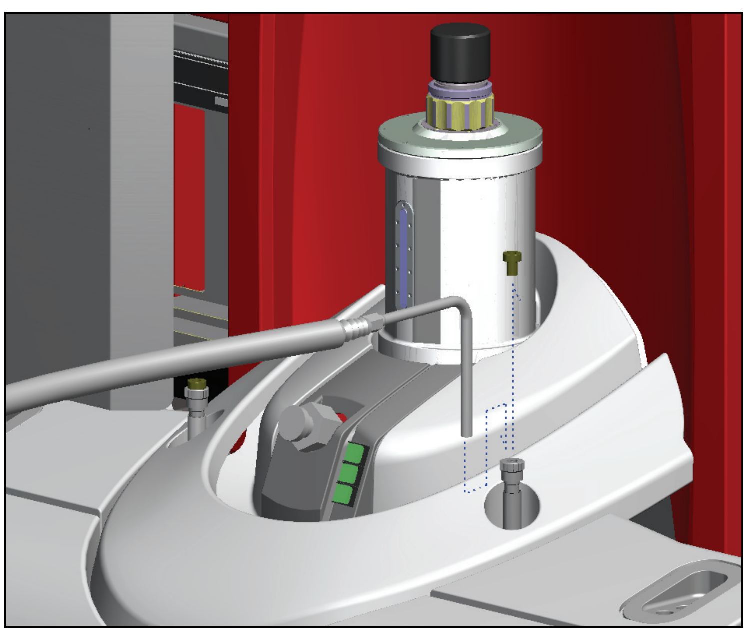


Figure 1-16. Filling liquid nitrogen.

3. Open the valve on the nitrogen storage dewar to start the transfer.

CAUTION: If during the start of the transfer the rear exhaust does not open within the first 30 seconds, then stop transferring and refer to Appendix A.3.10.

4. Transfer until the tank is full. Liquid nitrogen droplets will be expelled from the exhaust hose connected to the back of the MPMS SQUID VSM when the tank is full. The rubber hose exhaust should be visible to the operator when operating the system, in particular during transfers.
5. Close the valve on the nitrogen storage dewar. The nitrogen fill port and adapter will be frozen. Wait a few minutes until the hose and fill port warm up. Remove the hose and replace the plug in the nitrogen fill port.

CAUTION: Cold nitrogen gas/liquid might backstream out of the open port.

SERVICE NOTE: Inspect o-rings in the nitrogen inlet fittings once a month. Spare o-rings can be found in your utility kit in case they are damaged. Apply vacuum grease to keep them lubricated. Maintaining the nitrogen ports is important because it will prevent from ice plugging which can cause irreversible damage to the system.

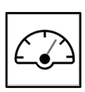
1.5.2 Fill the Liquid Helium Tank

Ramp the field to zero before starting transferring helium. Introducing warm gas into the dewar will cause the gas-cooled magnet to quench.

1. Start the helium fill utility:

Select the software menu item “Utilities > Helium Fill…”

OR

Press “fill” 

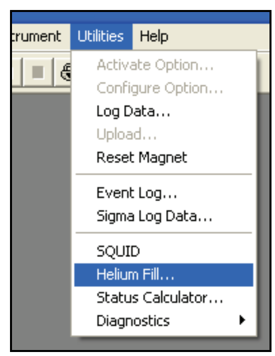


Figure 1-17. Utilities menu.

The helium fill status and graph will be displayed.

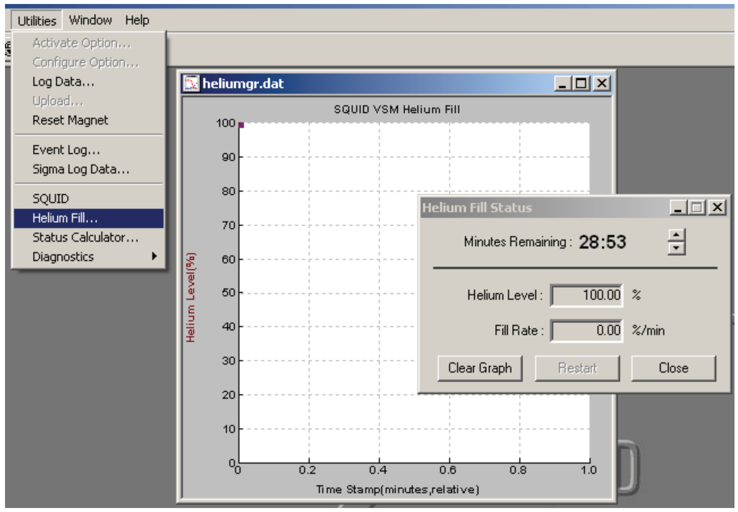
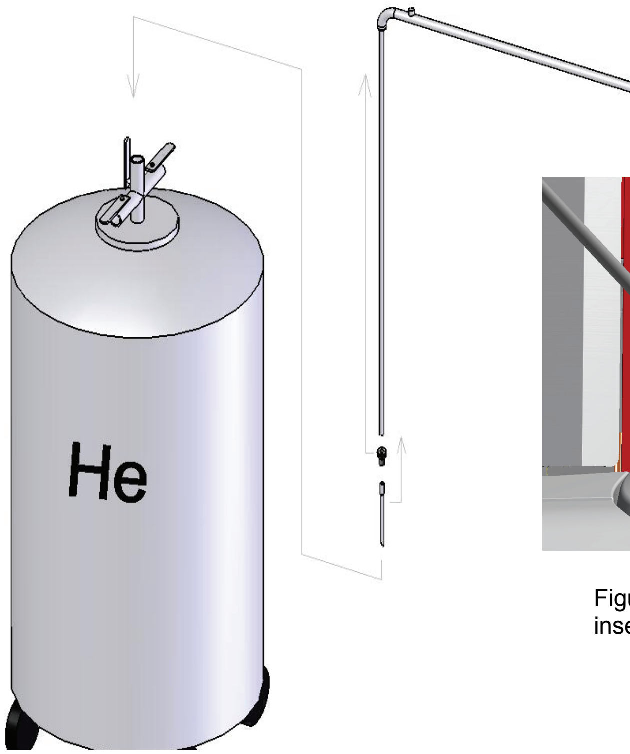


Figure 1-18. Helium fill utility.

2. Install attachments and insert the transfer line into the storage dewar:

   a. Make sure the appropriate extension for your storage dewar is on the long end of the helium transfer line—it should just reach the bottom of the storage dewar. Make sure the tube fitting is also on the long end of the transfer line.
   b. Vent excess pressure from the helium storage dewar. Then open the main valve on the helium storage dewar and insert the long end of the helium transfer line.
   c. Tighten the fitting onto the tube adapter. Then slowly lower the transfer line into the liquid helium until pressure builds up and gas begins flowing from the other end of the transfer line.
   d. Close the storage dewar safety relief valve so you may pressurize the storage dewar.



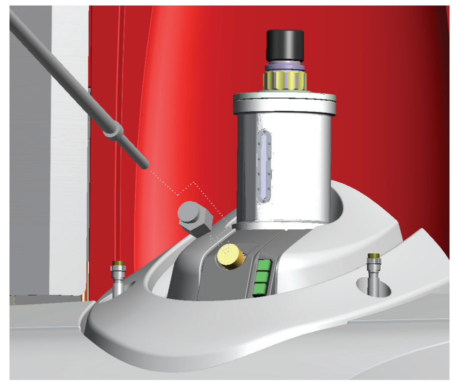


Figure 1-20. Helium transfer line insertion.

Figure 1-19. Liquid helium storage dewar.

3. Open the helium fill port on the SQUID VSM and rapidly insert the transfer line (Fig. 1-20), then screw the fitting tight.

CAUTION: Cold helium might backstream out of the open fill port.

4. Monitor the transfer as follows:
   a. Lower the transfer line all the way to the bottom of the storage dewar then raise it up about 2 cm (1 in.) to prevent transfer of particulate contamination that has settled on the bottom.
   b. Pressurize the storage dewar with helium gas and maintain a gas flow into the dewar of about 3 liters per minute. The fill rate should not exceed 2%/minute.
   c. Monitor the helium level in the software, and adjust the gas flow to the storage dewar if the transfer rate slows.
5. When the meter reads full (100% ~70 liters), stop pressurizing the storage dewar, open the safety relief valve on the storage dewar, and remove the transfer line from the SQUID VSM and quickly replace the cap on the fill port. Make sure cap is screwed on tight to avoid ice collecting and plugging of the transfer insert on the probe. Then remove the transfer line from the storage dewar and close the storage dewar main valve. Open the storage dewar pressure safety relief valve to ensure that no overpressure is building up inside the storage dewar.

1.6 Quick Reference: Buttons and Status Lights

The tables below describe the function and behavior of the three buttons and three status lights on the front of the SQUID VSM. The status lights are embedded within the buttons, but they function independently of the buttons.

Table 1.1 - Button Functions

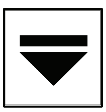
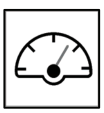
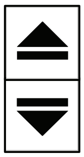
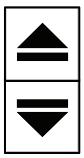
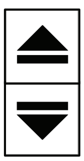
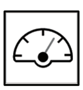
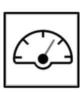
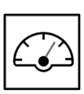
Button	Name	Used to…	Function when Pressed
	Eject	eject samples and open chamber	Vents sample chamber continuously with helium and moves sample transport to top of travel. Disabled when chamber is not at room temperature and when measurement is in process.
	Load	close chamber	Purges sample chamber with helium, leaves chamber evacuated
	Fill	start helium level monitor	Turns on helium level meter and starts the helium fill utility software. Same as selecting "Utilities > Helium Fill…"

Table 1.2 - Status Light States

Light	State	Color	Means
	on (solid)	green	Ready: chamber has been purged, no sequence is running
	blinking slowly	green	Busy: running sequence
		yellow	Busy: purging chamber
	blinking rapidly	yellow	Venting: you should replace the cap and press the load button to purge the chamber as soon as possible
		red	Error: see the MultiVu event log (“Utilities > Event Log…”)
	on (solid)	green	Status: last Helium level reading OK
		yellow	Status: last Helium level reading < 25% (~18 liters). OK to use system, but fill soon.
	blinking rapidly	red	Warning: last Helium level reading extremely low (< 10% ~7 liters). Fill the system as soon as possible. SQUID is not immersed in liquid helium (yields noisy measurements), inlet of flow controller is not immersed in liquid helium (yields loss of temperature control), magnet at risk (quench). System will shut down and recover once helium has been filled. Always fill both helium and nitrogen tanks
	blinking slowly	yellow	Busy: reading helium level meter

C H A P T E R 2

Software

2.1 Overview

In this chapter we describe MultiVu, the Windows™ software that coordinates the operation of the MPMS SQUID VSM hardware. MultiVu combines in a single user interface the basic instrument control, status reporting (section 2.2.5), data collection, graphing (section 2.4), sequence editing and sequence execution (section 2.3). In addition to these features, MultiVu also contains instrument utilities, diagnostics, and error reporting.

Start SQUID VSM MultiVu by double clicking the desktop icon:

MultiVu may be freely installed on additional computers. Once installed, use the desktop icon for running MultiVu in simulation mode, which works without SQUID VSM hardware attached. In simulation mode, MultiVu will not control the instrument, report the instrument status, collect data, or execute sequences. Simulation mode allows you to use the sequence editing and data graphing features of the software from different computers, and is not intended for use on the SQUID VSM computer.

When the MultiVu software is not running, the SQUID VSM goes into a hibernation state to prevent equipment damage. Most users will leave MultiVu running on the SQUID VSM computer at all times. MultiVu has a control to place the SQUID VSM in shutdown mode to conserve resources and still monitor the equipment status when it is not in use. Generally, it is not necessary to quit the MultiVu software.

2.1.1 Graphical User Interface

The main MultiVu window displays essential status information about the instrument and allows controlling the instrument with simple mouse click commands. The software also allows you to view two different types of files: sequence files (.seq) and data files (.dat.) Sequence files are used to automate the instrument operation. Data files are collections of time-stamped data recorded by the instrument. The commands available in the user interface change based on the type of file in the active window within MultiVu. Shortcuts to common commands are found in context-sensitive menus you access by right-clicking on a data file (graph) window or on a sequence file window.

2.1.2 Automation With Sequences

Instrument operation is automated with files called sequences. Sequences are similar to simple computer programs or scripts that execute linearly. Program branching and logic are not part of the MPMS SQUID VSM sequence language, but some looping operations are provided.

MultiVu contains an easy sequence editor where you double-click the command you want to insert into a sequence file, then fill in command parameters in a pop-up window. The editor allows typical Windows cut, copy, paste, and delete functions. The predefined sequence commands allow tremendous flexibility when automating the SQUID VSM operation.

For advanced users, the editor also allows access to the underlying script that is executed when a sequence is run, so that advanced programming techniques can be applied. Such advanced techniques are not necessary for most users.

MultiVu is also used to run, pause, lock, and abort sequences on the SQUID VSM and to view the status of sequences as they run.

2.1.3 Data Files and Graphing

Automation and the rapid data collection capabilities of the SQUID VSM allow large amounts of data to be saved to data files on the computer hard drive. Data files may be viewed in different formats, including graphs, tables, data records, and even raw text. The graph views may be extensively manipulated to help analyze data. Graph views may also be saved as templates for viewing other data so that common graph settings, such as data item selection, axis scaling, display range, and grid display may be applied in aggregate to any data file. The cells in table views may be copied and pasted into spreadsheets and graphing programs. However, the data file itself may not be altered or edited in MultiVu, except by adding data records and comments.

2.2 Graphical User Interface

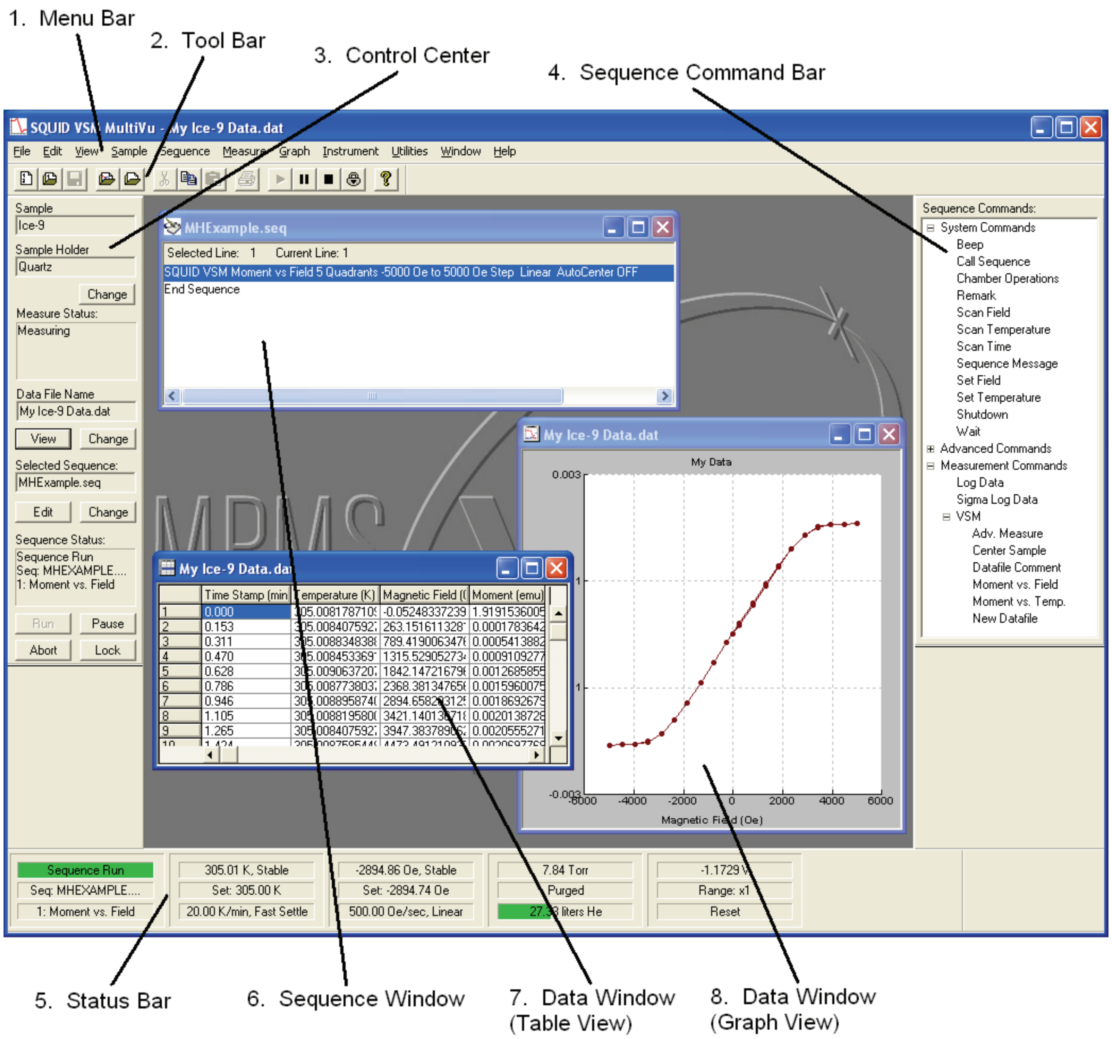


Figure 2-1. User Interface overview.

The main MultiVu window is shown above. The tool bar, control center, sequence command bar, and status bar may each be docked and un-docked by double-clicking any non-control region or by dragging them around the main window. They may also each be hidden using the “View” menu.

2.2.1 Menu Bar

The Menu Bar contains menus for accessing all fundamental software features.

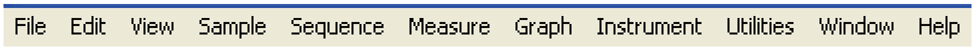


Figure 2-2. Main menu bar.

- **FILE** - File menu contains commands for opening, closing, and printing sequence files and data files; for saving sequence files, graph files and graph template files; for generating new sequence files; and for exporting data files to alternate formats.
- **EDIT** - Edit menu contains commands to edit sequence files. It is not shown unless a sequence window is selected.
- **VIEW** - View menu allows you to show and hide the various components of the MultiVu graphical user interface, and to open different types of data file windows (graph view, table view, record view, raw data view.)
- **SAMPLE** - Sample menu helps you install, remove, and locate samples and enter sample properties that are recorded in the data file.
- **SEQUENCE -** Sequence menu contains commands to control the execution of sequences and to access the underlying script for advanced sequence editing.
- **MEASURE -** Measure menu contains commands to control magnetic moment measurement, to change the data file where measurement data is recorded, to add comments to the data file, and to change the unit system used to record magnetic measurements.
- **GRAPH -** Graph menu helps you manipulate the appearance of data file graph windows.
- **INSTRUMENT -** Instrument menu contains commands to control the temperature, magnetic field, and vacuum state of the sample chamber, and to put the instrument into shutdown mode when unused. For more information about the modes of instrument operation, see Chapter 3: Hardware.
- **UTILITIES -** Utilities menu contains commands to activate and configure instrument options, and to help maintain and troubleshoot the instrument, including filling the helium tank, tuning the SQUID, logging diagnostic data, and viewing error messages. (See appendix B for more information.)
- **WINDOW -** Window menu helps arrange the open windows in MultiVu.
- **HELP** - Help menu contains version and serial number information about the MPMS SQUID VSM, and access to the user documentation.

2.2.2 Tool Bar

The Tool Bar contains shortcuts for common menu items.

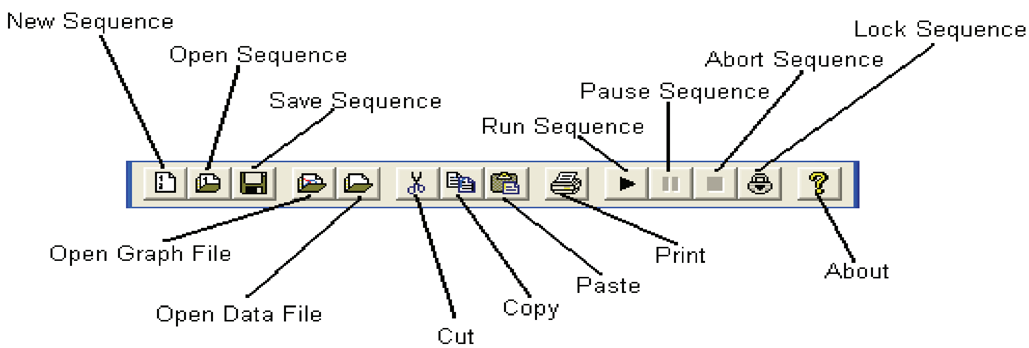


Figure 2-3. Tool bar.

2.2.3 Control Center

The Control Center (Fig. 2-4) provides an overview of the currently installed sample name, selected data file name, selected sequence file name, measurement status, and sequence execution status. In addition, there are buttons to provide quick access to change the sample; change the data file; change the selected sequence; edit, run, pause, abort, or lock the selected sequence; and view a graph of the current data file.

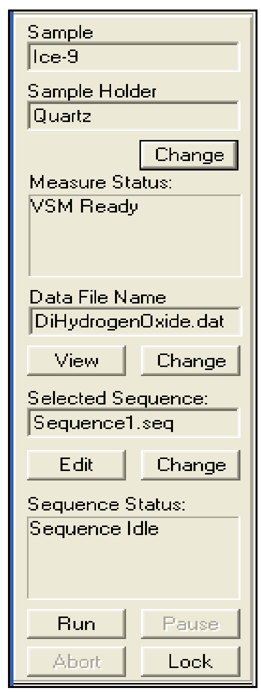


Figure 2-4. Control center.

2.2.4 Sequence Command Bar

The Sequence Command Bar (Fig. 2-5) is shown when you edit sequences. You can toggle its display by selecting the "View > Sequence Command Bar" entry while editing a sequence. It contains commands you may insert into the sequence. Commands are organized in a tree structure. Click "+" and "-" to expand or collapse tree branches. Double click commands to insert them into the sequence. More on sequences is found in the section below.

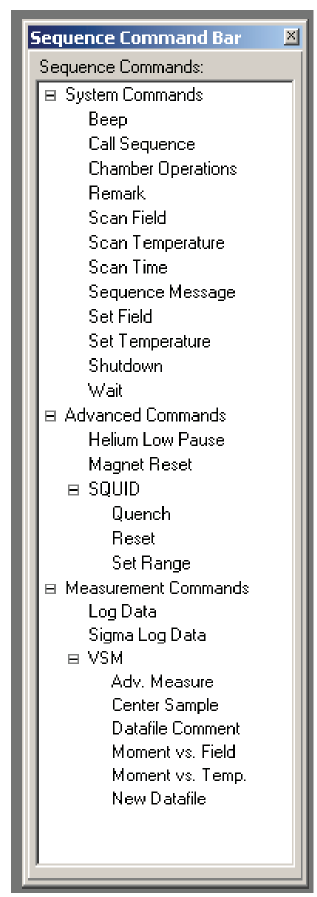
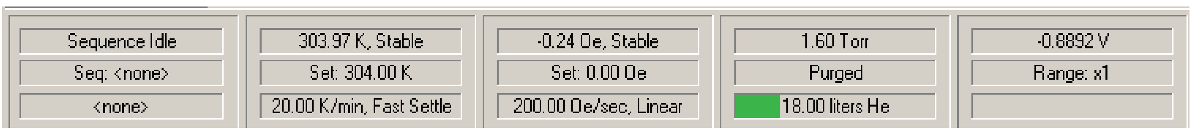


Figure 2-5.
Sequence command bar.

2.2.5 Status Bar

The Status Bar contains information about the status of the instrument. The color of the helium tank level reading changes to yellow, then red when the helium level drops too low. The length of the colored bar indicates the fill level. Click on the panels in the status bar as shortcuts to basic instrument control (such as temperature, magnetic field, and chamber vacuum state) and SQUID diagnostics.



Sequence:
- -status
- -file name
- -execution line

Temperature:
- -present state
- -setpoint
- -rate and mode

Magnetic Field:
- -present state
- -setpoint
- -rate and mode

Sample Chamber:
- -pressure
- -state

Helium Tank:
- -approximate volume remaining

SQUID:
- -reading
- -range

Figure 2-6. Status bar.

2.2.6 Sequence Window

The Sequence Window is used for editing sequences. More than one sequence window may be open at a time. The *selected* sequence is always the sequence in the last sequence window that was active (clicked). The control center reports the selected sequence file name. The selected sequence is the sequence into which commands will be inserted when selected with the sequence command bar. The selected sequence is also the sequence that executes when "Run Sequence"  is selected, when the control center "Run" button is selected, or when the menu command "Sequence > Run" is selected.

2.2.7 Data Window (Table View)

The Data Window (Table View) shows a data file in table format. Each row in the table represents a single data record. A data record consists of several data items collected simultaneously, such as sample temperature, magnetic field, and magnetic moment. You may view a single data record in its own window by double-clicking any record in the table view.

Cells and groups of cells in the table view can be selected, copied, and pasted into other spreadsheets and graphing programs for additional analysis using the standard Microsoft Windows™ copy and paste features.

2.2.8 Data Window (Graph View)

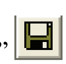
The Data Window (Graph View) shows a data file in graph format. Each point on the graph represents a single data record. A data record consists of several data items collected simultaneously, such as sample temperature, magnetic field, and magnetic moment. You may view a single data record in its own window by double-clicking any record in the graph view. The record view and graph view are linked, so that the highlighted point on the graph view corresponds to the record shown in the record view.

The appearance of the graph view may be manipulated extensively and templates may be applied to the graph view so that you do not need to set each graph characteristic individually. To learn more about manipulating the graph view, read the section below “Graphing Data Files.”

2.3 Sequences

Operation of the SQUID VSM can be automated using sequence files. Sequence files are like simple computer programs instructing the instrument to carry out a number of operations in a predetermined order. Operations typically found in a sequence file include changing the sample’s temperature, changing the magnetic field, and measuring the sample’s magnetic moment. Additionally, commands may be placed in sequences to log diagnostic data, center the sample, record a comment in the data file, begin recording data to a new data file, generate a message on the computer screen, and even run another sequence.

2.3.1 Editing Sequences

- Create a new sequence file by clicking “New Sequence”  or selecting the menu item “File > New Sequence.”
- Open an existing sequence file for editing by clicking “Open Sequence”  or selecting the menu item “File > Open… > Sequence.” You will be prompted to locate the file you want to open.
- The top of the sequence window shows the sequence file name. An asterisk symbol (*) after the file name means the sequence has changed since last time the file was saved. Sequences must be saved before you run them.
- Save a sequence file by clicking “Save Sequence”  or selecting the menu item “File > Save.” If not already assigned, you will be prompted for a location and file name.

- Add a command to the selected sequence by double-clicking the command in the sequence command bar. The new command is inserted above the highlighted command in the selected sequence window. You will first be presented with a pop-up window to fill in command-specific information. Then a summary of the command will appear in the sequence window. More detail about each available sequence command is found at the end of this section.
- Remove and reorganize sequence commands by highlighting them in the sequence window and using the “Cut,” “Copy,” “Paste,” “Delete,” and “Undo” commands in the “Edit” menu.
- Disable commands in a sequence file without deleting them using “Edit > Disable.” Enable a disabled command with “Edit > Enable” (Fig. 2-7). A disabled command will be skipped during sequence execution. Disabled commands are preceded by an exclamation mark symbol (“!”) in the sequence window and are changed from black text to gray text. With this feature you may decide to execute or not execute some commands just prior to run time based on the immediate circumstances. For example, you may wish to disable a series of commands that fall outside the meaningful measurement range for certain samples.

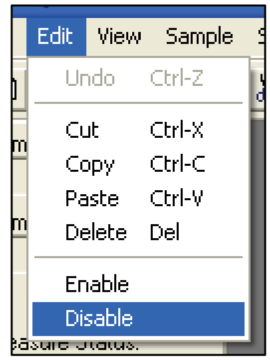


Figure 2-7. Disabling sequence commands.

- All of the commands found in the “Edit” menu are duplicated in a pop-up menu when you right-click any sequence window.

2.3.2 Advanced Script Editing (Macros)

Before running sequences, MultiVu automatically compiles sequence files into a Visual Basic for Applications type of script. Users with programming experience may compile this script without running the sequence, and may then edit the script directly and run the edited script (macro). However, Quantum Design cannot certify the instrument behavior when it is automated with user-designed scripts. Many checks and safeguards are bypassed when running a sequence script directly. Novice users should not use this feature. Advanced users with programming experience should use this feature with caution.

- Compile and edit the sequence script using the menu commands “Sequence > Advanced > Compile Macro” and “Sequence > Advanced > Edit Macro.” A Sax Basic editor window will appear.
- Run the script with the menu command “Sequence > Advanced > Run Macro.” You will be prompted to locate the script file to run. Or you may run the script by clicking the “Start/Resume” button  in the Sax Basic editor window.
- Find more information about the Sax Basic editor and scripting language by right-clicking in the Sax Basic editor window and selecting “Help > Editor Help” and “Help > Language Help”.

2.3.3 Running Sequences

- If the sequence file you want to run is not open, you need to open it. Open an existing sequence file by clicking “Open Sequence” or selecting the menu item “File > Open… > Sequence.” You will be prompted to locate the file you want to open.
- To execute a sequence, it must be the selected sequence. If more than one sequence window is open, make sure the selected sequence is the sequence you want to execute.
- Execute the selected sequence by:
  - Selecting “Run Sequence”

    OR
  - Selecting the menu command “Sequence > Run.”

    OR
  - Selecting the “Run” button on the control center.
- The status bar and the control center both display the status of the sequence execution. The sequence window also highlights the line currently being executed in green.
- The toolbar, “Sequence” menu, and command center all provide the ability to pause () and abort () sequence execution also. When paused, the sequence window highlights the active line of the sequence in yellow.

2.3.4 Locking Sequence Execution

- When the sequence execution is locked, a sequence cannot be run, paused, or aborted without first unlocking sequence execution. You cannot exit MultiVu when sequence execution is locked either.
- No key or password is required to lock or unlock sequence execution. The feature is intended only to prevent careless and accidental interference with instrument operation. It is not a security device against malicious behavior.
- The instrument does not need to be running a sequence to lock sequence execution.
- Lock sequence execution by clicking “Lock Sequence” . Or select the menu item “Sequence > Lock,” or click the “Lock” button on the control center. Type in your name and additional information so other users know why the sequence is locked, then click the “Lock” button. All run, pause, and abort controls in the graphical user interface are disabled.

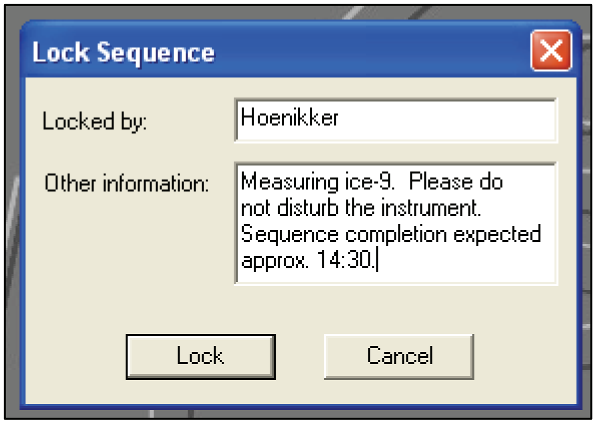


Figure 2-8. Locking/unlocking sequences.

- Unlock the sequence by clicking “Lock Sequence”  again. Or select the menu item “Sequence > Unlock,” or the “Unlock” button on the control center. Then click the “Unlock” button on the popup dialog, which displays the name and message of the person who locked sequence execution.

2.3.5 Sequence Commands

The following sequence commands can be used in sequences. Option software purchased with the MPMS SQUID VSM may provide additional sequence command not listed here.

2.3.5.1 SYSTEM COMMANDS:

- **Beep** – Causes the computer to make the Windows default beep sound.
- **Call Sequence or Script** – Suspends execution of selected parent sequence file and begins execution of specified child sequence file or script file. When execution of specified child sequence file or script is complete, execution of selected parent sequence will continue with the next line.
- **Chamber Operations** – Changes state of the sample chamber atmosphere. (Fig. 2-9)
- **Remark** – Serves as a message, comment, or visual break for the user only. Does nothing when executed in a sequence file.
- **Scan Field** – Creates a program loop for executing repeated commands at user-defined magnetic field increments. All commands between the “Scan Field…” line in the sequence and the “End Scan” line in the sequence will be

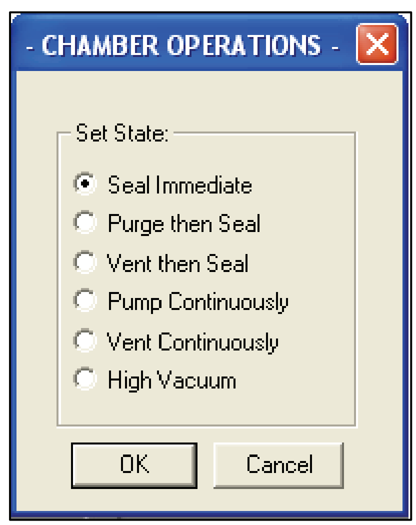


Figure 2-9. Chamber operations menu.

be repeated at each magnetic field specified by the scan field command. Set the initial and final fields and the scale on which the field increments should appear uniform (linear, H^2, H^1/2, 1/H, log(H).) Also set the total number of field steps. (For uniform linear spacing, you may alternatively set the field increment.) Finally, specify the rate and approach mode used by the magnet controller to achieve each set point:

- *Linear*: Controller will drive directly to each field and attempt to maintain the specified charging rate as closely as possible until each set point is reached. At each set point the field will stabilize until the commands within the loop are completed. A small amount of field overshoot can occur in this mode.
- *No Overshoot*: Controller will drive 80% of the way to each set point at the desired rate, and will then slowly step up (or down) to the set point to avoid any field overshoot. At each set point the field will stabilize until the commands within the loop are completed. This is intended for use with highly field-hysteretic samples.
- *Oscillate*: Controller will intentionally overshoot each set point by 70% of the total field change, at the desired rate, and will then oscillate into the set point field in smaller and smaller overshooting steps. At each set point the field will stabilize until the commands within the loop are completed. This is intended to eliminate flux motion in the superconducting magnet windings, yielding a very stable magnetic field and more stable SQUID operation. True zero field is best achieved using oscillate mode.
- *Sweep*: Controller will drive directly from the initial field to the final field without stopping. Each time a field increment defined by the command is reached, the commands inside the scan field loop will be executed, but the field will continue to change while they execute.

- **Scan Temperature** – Creates a program loop for executing repeated commands at user-defined temperature increments. All commands between the “Scan Temperature…” line in the sequence and the “End Scan” line in the sequence will be repeated at each temperature specified by the scan temperature command. Set the initial and final temperatures and the scale on which the spacing of the temperature steps should appear uniform (linear, 1/T, log(T).) Also set the total number of temperature steps. (For uniform linear spacing, you may alternatively set the temperature increment.) Finally, specify the rate and approach mode used by the temperature controller to achieve each set point:
  - *Fast*: Controller will drive directly to each temperature and attempt to maintain the specified sweep rate as closely as possible until each set point is reached. At each set point the temperature will stabilize until the commands within the loop are completed. A small temperature overshoot can occur in this mode.
  - *No Overshoot*: Controller will drive to each set point at the desired rate until it is less than 30 seconds from achieving the setpoint, and will then slow the rate significantly to avoid temperature overshoot. At each set point the temperature will stabilize until the commands within the loop are completed. This is intended for use with highly temperature-hysteretic samples.
  - *Sweep*: Controller will drive directly from the initial temperature to the final temperature without stopping. Each time a temperature increment defined by the command is reached, the commands inside the scan temperature loop will be executed, but the temperature will continue to change while they execute.

- **Scan Time** – Creates a program loop for executing repeated commands at user-defined time increments (or immediate repetitions with no time increment.) All commands between the "Scan Time…" line in the sequence and the "End Scan" line in the sequence will be repeated at each time specified by the scan time command. Set the total time in seconds and specify whether the spacing of events should be uniform in time or logarithmic in time. And specify the number of steps. This is the number of times the loop will be repeated. (For uniform spacing in time, you may alternatively specify the time increment.) If the total time is set to zero seconds, then the number of steps defines how many times the loop will be repeated in rapid succession.
- **Sequence Message** – Displays a message on the computer screen and pauses sequence execution until the message is acknowledged or until a timer expires. If the computer is set up with internet access and access to a mail server, a message can also be emailed with attachments such as data files.
- **Set Field** – Sets the instrument's magnetic field. Specify the field, the charging rate, and the approach:
    - *Linear*: Controller will drive directly to the field and attempt to maintain the specified charging rate as closely as possible. A small amount of field overshoot can occur in this mode.
    - *No Overshoot*: Controller will drive 80% of the way to the set point at the desired rate, and will then slowly step up (or down) to the set point to avoid any field overshoot. This is intended for use with highly field-hysteretic samples.
    - *Oscillate*: Controller will intentionally overshoot the set point by 70% of the field change, at the desired rate, and will then oscillate into the set point field in smaller and smaller overshooting steps. This is intended to eliminate flux motion in the superconducting magnet windings, yielding a very stable magnetic field and more stable SQUID operation. True zero field is best achieved using oscillate mode.

  Notice that sequence execution continues with the next command in the sequence as soon as the field is set, *not* when the field set point is achieved. To wait for a stable magnetic field before executing the next command, use the **Wait** command.
- **Set Temperature** – Sets the sample temperature. Specify the temperature, the rate, and the mode:
    - *Fast Settle*: Controller will drive directly to the temperature and attempt to maintain the specified rate as closely as possible until the set point is reached. A small amount of temperature overshoot can occur in this mode.
    - *No Overshoot*: Controller will drive to each set point at the desired rate until it is less than 30 seconds from achieving the setpoint, and will then slow the rate significantly to avoid temperature overshoot. This is intended for use with highly temperature-hysteretic samples.

  Notice that sequence execution continues with the next command in the sequence as soon as the temperature is set, *not* when the temperature set point is achieved. To wait for a stable temperature before executing the next command, use the **Wait** command.
- **Shutdown** – Places the instrument in shutdown mode to conserve resources. The motor is powered down, the field is set to zero and the magnet controller is turned off, the heaters are turned off, and the valve driving the cooling power is closed.
- **Wait** – Waits for specified conditions to be achieved, then delays a specified amount of time before continuing with sequence execution. Conditions that can be specified to wait for are temperature stability, field stability, stepper motor position (not currently in use,) and chamber state. This command is usually used immediately after another command in order to make sure the desired outcome of the first command is achieved before proceeding. For example:

Set Temperature 77K at 10 K/min. Fast Settle

Wait For Temperature. Delay 10 secs, No Action

Measure Moment vs. Field…

This sequence will wait 10 seconds after the instrument has achieved temperature stability at 77K before beginning a series of moment measurements at various magnetic fields. Specify, also, what the instrument should do if an error occurs while waiting for the specified conditions (no action, abort sequence, or shutdown instrument.)

2.3.5.2 ADVANCED COMMANDS:

- **Helium Low Pause** – Pauses sequence execution if the liquid helium level drops below a specified reading on any normal helium level update. This command is useful in very long sequences, when execution might take place over a period when nobody is present to monitor the helium level. For safety reasons, any running sequence will be automatically stopped and the system shutdown when the helium level gets below 10% (~7 liters). (see section 1.6)
- **Magnet Reset** – Drives the magnet to zero field and applies heat to the superconducting magnet windings to remove trapped magnetic flux. (See chapter 4 for application considerations.)
- **SQUID** – Allows direct control of the instrument's SQUID. It is not usually necessary to utilize these controls because the instrument automation already optimizes SQUID operation.
    - **Quench** – Applies heat to the SQUID or to the SQUID input (superconducting detection coil circuit) to eliminate standing currents.
    - **Reset** – Holds the SQUID in reset for a specified amount of time, applying heat directly to the SQUID to return the SQUID output to nearly zero volts.
    - **Set Range** – Sets the SQUID head gain manually. Overrides any auto-ranging that may be in effect.

2.3.5.3 MEASUREMENT COMMANDS

- **Log Data** – Records specified diagnostic data to a data file at a specified rate. No size limit is imposed on the data file generated, so the data file can get extremely large and difficult (or slow) to process. See appendix B for more information on logging diagnostic data.
- **Sigma Log Data** – Records specified diagnostic data to a data file at a specified rate. This command is similar to the Log Data command, except that it allows you to record statistics such as a running average and standard deviation for each data item, in each data record. It may generate large data files with large header sections. See appendix B for more information on logging diagnostic data.
- **VSM**
    - **Adv. Measure** – Creates a sequence step specifying all necessary parameters (see tabs below) to run a measurement.

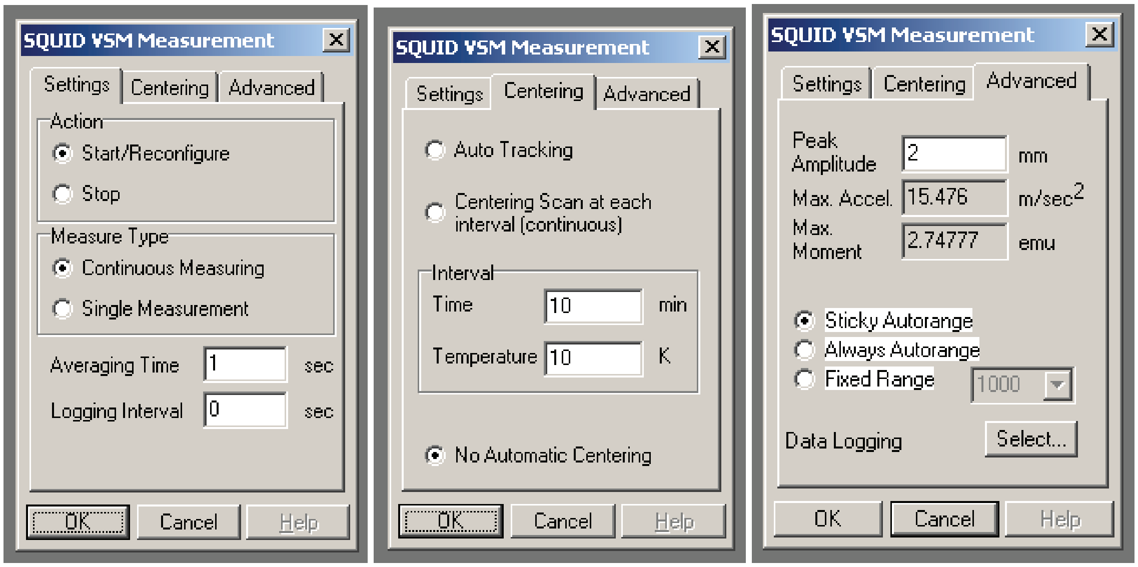


Figure 2-10. SVSM measurement parameters interface.

- Specify "Continuous Measuring" for a continuous stream of data, or "Single Measurement" to collect a single data point.
- Specify the averaging time and the logging interval (for continuous measurement stream).
- On the "Centering" tab, specify which automatic centering method is used to keep the sample centered.
- On the "Advanced" tab, you may also set the vibration amplitude and SQUID range settings. 2 mm is the default vibration amplitude. The instrument achieves highest sensitivity with vibration amplitudes greater than 2 mm. (See chapter 4 for vibration amplitude considerations.)
- Set the instrument temperature or magnetic field to vary those parameters as desired.

○ **Center Sample** – Allows you to reposition the sample for accurate measurements, and to change the automatic centering setting. (Also allows advanced operations: moving the sample to any specified location, motor touchdown and motor home.) If your sample begins the sequence well-centered and automatic centering is enabled, this command may not be necessary.

○ **Datafile Comment** – Puts a comment in the present data file, along with a time stamp. The comment and time stamp constitute a single data record in the data file.

○ **Moment vs. Field** – Performs a series of magnetic moment measurements at specified magnetic fields (see also chapter 1.) You specify the maximum and minimum fields and one intermediate field (typically zero field) and the shape of the field ramps to perform, for example, four-quadrant or five-quadrant hysteresis loops. The series may begin and end on either the maximum, minimum, or intermediate field. You also control the rate of field change and whether the field stabilizes for each measurement or continues changing (sweeps) while the measurement is performed. You specify the VSM measurement averaging time and the data spacing in field (H) (uniform in H, H^2, H^1/2, 1/H, or log(H)) and the number of data points per two-quadrant ramp. Alternatively, you may also specify "continuous measuring," in which case the instrument will record data as quickly as possible, with your averaging time, while the field sweeps are performed. You may also repeat multiple measurements at each magnetic field. The pop-up window displays the approximate magnetic fields where measurements will be performed with your settings, and the estimated total time to perform the measurement series. An "advanced" tab also allows you to change the automatic centering setting, SQUID ranging, and diagnostic data logging, as well as a wait time at each field prior to commencing measurement, and the magnet approach mode (linear, oscillate, no overshoot—see set field command below) for each field. The "advanced" tab also allows you to change the measurement amplitude and frequency. (See chapter 4 for application considerations.)

- **Moment vs. Temp. –** Performs a series of magnetic moment measurements at specified temperatures (see also chapter 1.) You specify the start and end temperature, and the rate of temperature change. You also control whether the temperature stabilizes for each measurement or continues changing (sweeps) while the measurement is performed. You specify the VSM measurement averaging time and the data spacing in temperature (T) (uniform in T, T^2, T^1/2, 1/T, log(T)) and the total number of data points. Alternatively, you may also specify "continuous measuring," in which case the instrument will record data as quickly as possible, with your averaging time, while the temperature sweep is performed. You may also repeat multiple measurements at each temperature. The pop-up window displays the approximate temperatures where measurements will be performed with your settings, and the estimated total time to perform the measurement series. An "advanced" tab also allows you to change the automatic centering setting, SQUID ranging, and diagnostic data logging, as well as a wait time at each temperature prior to commencing measurement, and the temperature approach mode (fast settle, no overshoot—see set temperature command above) for each temperature. The "advanced" tab also allows you to change the measurement amplitude and frequency. (See chapter 4 for application considerations.)
- **New Datafile** – Changes the data file being used to record data (Fig. 2-10). You must specify a path and file name for the new data file. In addition, specify whether data should be written to a new version of the data file or appended to the end of the existing file if the specified file already exists. If you select "Create New File/Version" and a file with the specified path and file name already exists, the data will be written to a new file with the specified file name plus a five digit number appended to the end of the file name as a unique identifier, such as "MyDataFile_00001.dat," "MyDataFile_00002.dat," etc.

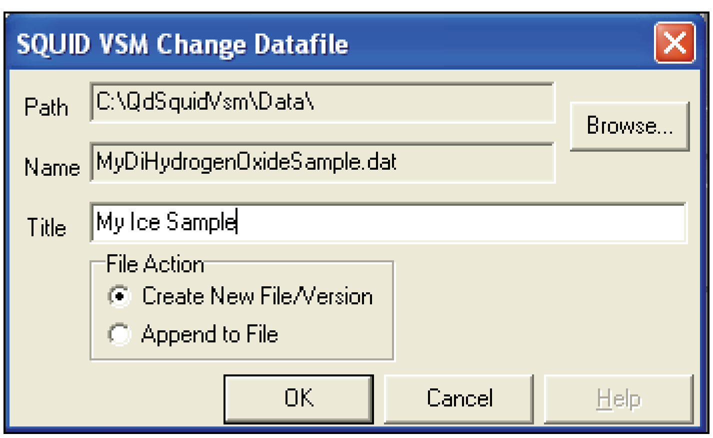


Figure 2-11. Creating new datafile.

2.4 Graphing Data Files

The graph view is the default for viewing data files. Any data file with one or more data records may be opened in a graph view. The appearance of graph views may be manipulated extensively to aid data analysis.

- Open a data file to graph by clicking "Open Data File"  or by selecting "File > Open > Datafile." Or on the control center click "View" to open the current data file.

2.4.1 Data Selection and Plot Axes

- Change the plot axes by selecting "Graph > Data Selection…" or right-click in an open graph window and select "Data Selection…"

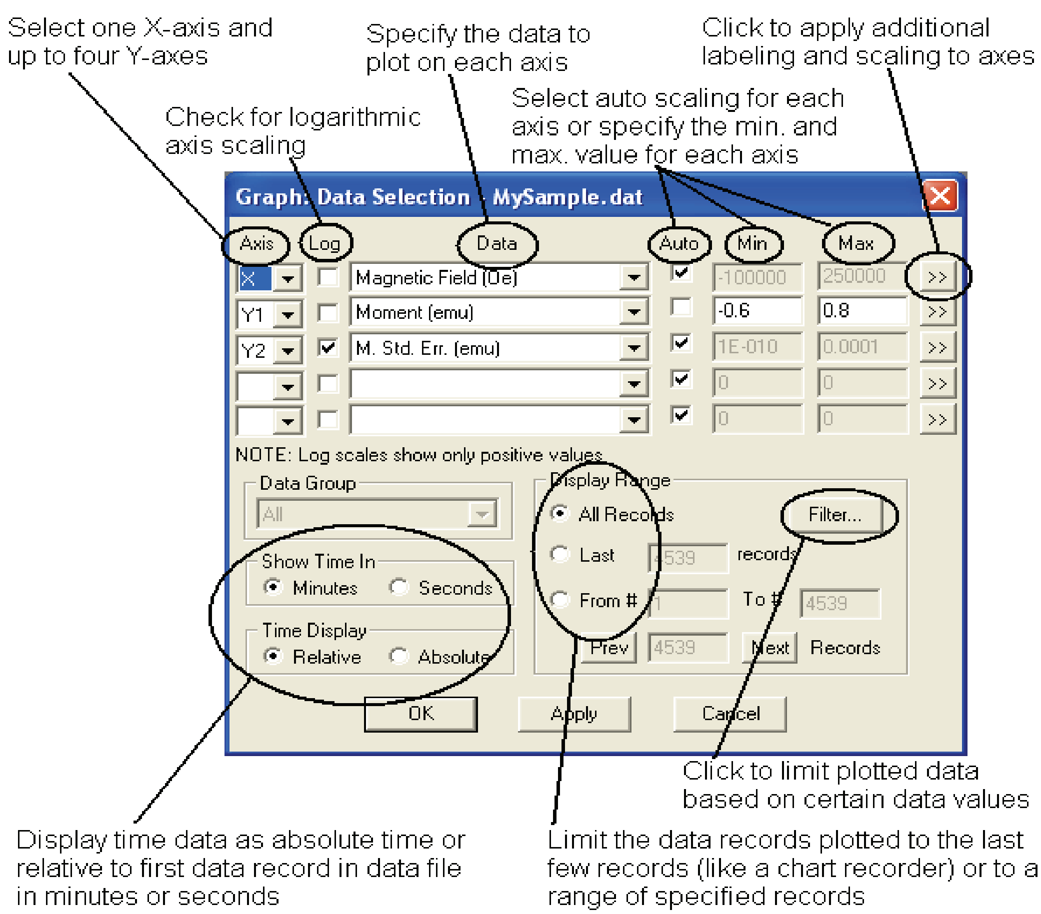


Figure 2-12. Graphing parameters interface.

- The axes may be assigned any label for which data exists in the data file.
- Up to four vertical axes may be displayed on separate plots in the same graph window, but all plots in the graph window must share the same horizontal (x) axis.
- When auto-scaling is selected for all axes, the plot axes will be rescaled each time new data is written to the data file so that all data is displayed. This helps optimize data viewing as data is being collected.
- Quickly auto-scale the axes by right-clicking in the graph window and selecting "Auto Scale All Plots."
- Use the ">>" button to add custom axis labels and to change the axis scaling multiplier by factors of 10.
- Use the "Filter…" button to plot only records with data that falls within a specified range. For example, specify a range for moment standard error to hide data with a large standard error (i.e. noisy data.)
- The "OK" button applies the changes to the graph window and closes the Data Selection dialog. The "Apply" button applies the changes to the graph window without closing the dialog. The "Cancel" button closes the Data Selection dialog without making any changes to the graph window.

2.4.2 Plot Appearances

- Change the appearance of each plot by selecting "Graph > Appearance > Plot 1" (or "… Plot 2," etc.) or right-click the plot you want to change and select "Appearance."
- Turn horizontal and vertical grid lines on and off with this dialog.
- Choose between markers on each data record, lines between each data record, or both markers and lines. The lines shown can be limited to only those in the positive or negative x-quadrant direction using the ">>" button.
- Check "Apply to All Plots" to apply the appearance settings to all plots in the graph window.

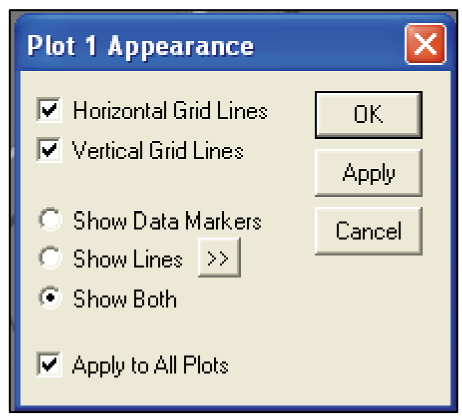


Figure 2-13. Plot appearance interface.

2.4.3 Templates and Graph Files

Groups of graph view settings may be saved in template files (.tpl) and applied to other data files so the graph view of each data file will look similar. The settings saved in template files include all settings in the Data Selection and Plot Appearance dialogs. This feature helps view many different data files in the same graphic format.

A graph file (.gph) is a template file that MultiVu automatically applies to the data file (.dat) *with the same name* whenever the data file is opened in graph view. You may save template files as graph files (.gph), but it is not necessary to do so. MultiVu automatically saves a graph file for each data file whenever all graph views are closed so that the graph view of the data file will look the same next time it is opened.

- To save a template file, format the graph view as desired, then:
  - Select “File > Save Template”
    OR
  - Select “Graph > Save Template”
    OR
  - Right-click the graph view window and select “Save Template”

Then specify the path and file name for the template file. (Specify “Graph Files (.gph)” in the “Save as type:” box to save the template as a graph file. Be sure the file name you specify matches the data file you want the graph applied to in this case.)

- To apply a template to a data file, select “Graph > Apply Template…” (or right-click the graph view and select “Apply Template…”) Then locate the template or graph file you want to use.
- “Graph > Restore Graph” applies the settings from the graph file to the graph window. This will undo any settings changed since last time the graph file was saved.
- “Graph > Default Graph” sets the axes to Moment and Time Stamp, with auto-scaling on both axes.

2.4.4 Exporting Data Files

- Many spreadsheet programs can read the standard .dat files SQUID VSM MultiVu generates. They have a comma-delimited format. But the files contain additional header information and may contain diagnostic data that complicates your data analysis or communication with collaborators. To export data files, or subsets of the data in a data file, to an alternate file format use “File > Export…” This feature generates data files from the data you specify in several formats that most graphing and spreadsheet programs understand.

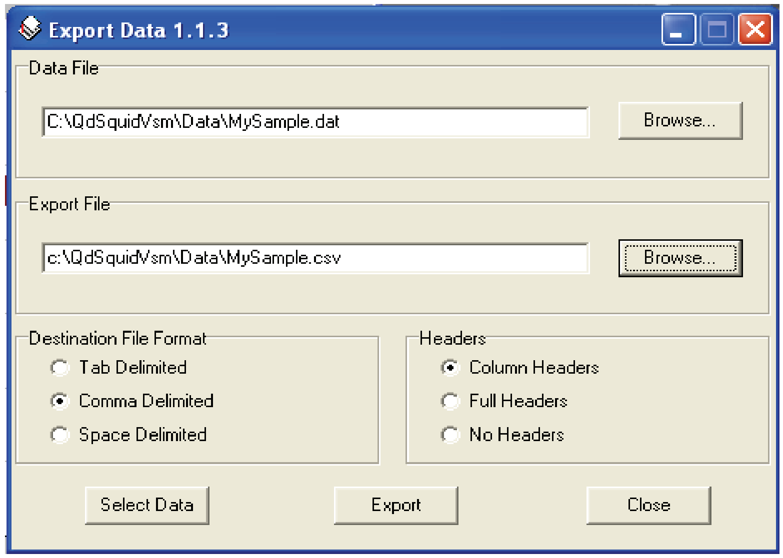


Figure 2-14. Exporting data.

- Make sure "Data File" specifies the file you want to export.
- Designate the path and file name of the new file to write under "Export File."
- Under "Destination File Format," choose which character will separate data items in the new file: tab, comma, or space.
- Choose whether you want column headers (column labels), full headers, or no headers.
  - With full headers, the file format must be comma delimited. All the header information at the beginning of the data file will be written to the new file, including sample properties, software revision, and other information used by MultiVu. This header information will appear at the beginning of the exported data file and may not be easily imported by other programs. This option is used to keep the existing file format but export a subset of the data.
- Click "Select Data" to specify which data items to export.
  - Available data items are listed on multiple tabs.
  - Check the box next to data items to write them to the export file.
  - Specify the order of the data columns in the export file by entering numbers in the "Col Order" boxes.
  - To exclude data that falls outside a certain range, check the "Select Range" box and specify the range of data to keep.
- Click "Export" to write the selected data to the new file. You will see a confirmation dialog when the operation is complete.

C H A P T E R 3

Hardware

3.1 Overview

In this chapter we describe the SQUID VSM hardware. The goal is to provide enough details to help understand how the system is working. In section 3.2 we give a general introduction on the system architecture and detail the power distribution, the module bay tower and the dewar setup. In the following sections we provide insight into the heart of the instrument: the temperature control (section 3.3) allowing to vary the temperature between 1.8 and 400 Kelvin, the magnetic field control (section 3.4) generating fields from -7 to 7 Tesla, the motion control (section 3.5) oscillating the sample in field, the SQUID detection system (section 3.6) sensing the induced signal collected by the pick-up coils and finally the facilities controlling the chamber atmosphere (section 3.7) and the cryogen level (section 3.8). Additional hardware such as the sample rod is described in section 3.9.

3.2 System Setup

The SQUID VSM hardware is primarily contained in a two-part metal cabinet. The upper half of the cabinet contains a computer running the MultiVu software and the instrument's modular electronics. The lower half of the cabinet contains a cryostat: liquid helium and liquid nitrogen tanks and a cryogenic insert that hangs into the helium tank. The VSM head, or motor, is mounted on top of the cryogenic insert. The lower end of the insert contains the shielded SQUID, superconducting magnet, and associated hardware. The cryostat is mounted on vibration isolation springs and magnetically shielded to confine magnetic fields greater than 5 Gauss to the interior of the cabinet. The lower half of the cabinet also contains storage for sample rods.

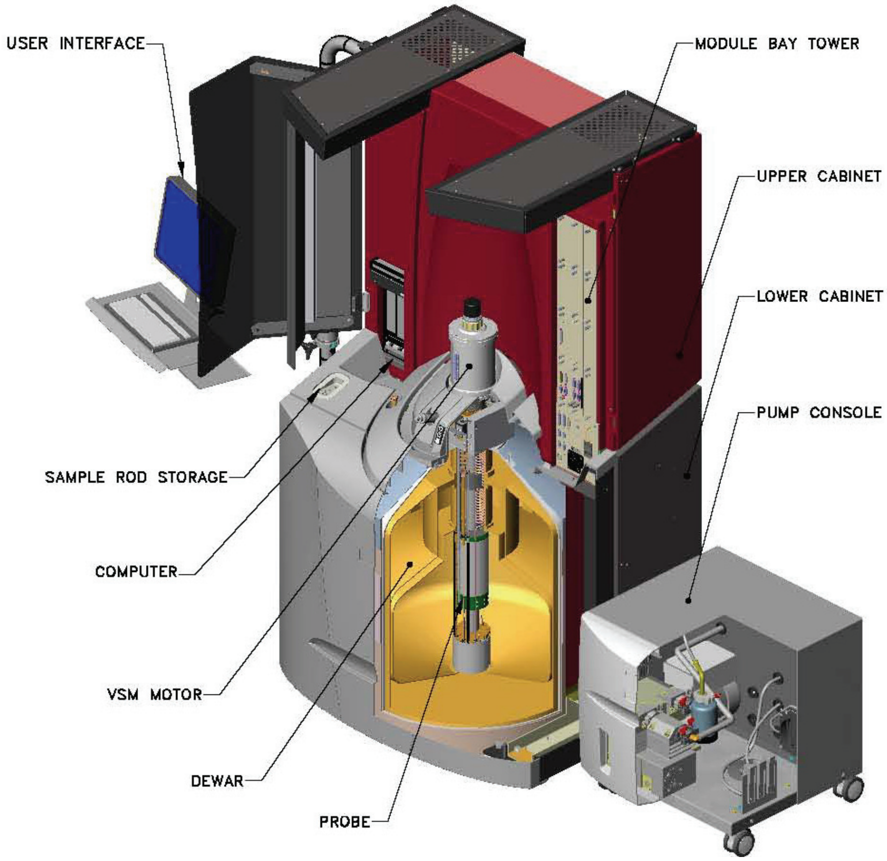


Figure 3-1. General System Setup

The entire cabinet is mounted on casters to simplify relocation, and has leveling mounts in the front to stabilize the instrument in its final destination. A number of cosmetic panels hide a large amount of the equipment, but may be removed to service the equipment or to fit the instrument through a standard 91 cm doorway.

3.2.1 System Power Distribution

All system electronics are powered from a single power cord, and may be turned off with a single switch, except on EverCool-equipped instruments. (In EverCool-equipped instruments, a compressor is required which is powered by a separate grounded 3-phase power cord.) The instrument is rated for single phase 200-230 VAC 50/60 Hz, and will draw 10A. The power distribution unit on the cabinet has two grounded outlets for connecting auxiliary hardware (the computer monitor and the pump cabinet) and there are dedicated outlets inside the cabinet for distributing power to the equipment. It is not recommended to use any of these outlets to power user equipment. The power distribution diagram can be found in Appendix D.3.

CAUTION: The instrument must be plugged into a simple phase IEC 60309 outlet (16A/250 V) fused at 16A. (See Appendix D.2.2 for more details).

Always power the system down before unplugging the power cord.

On the system side, the power cord is secured to the power distribution unit by a clamp. Do not tamper with this setup unless specifically instructed by qualified Quantum Design representatives. Risk for irreversible system damage and electrical shock hazard.

The main power switch has a resettable breaker rated at 10A.

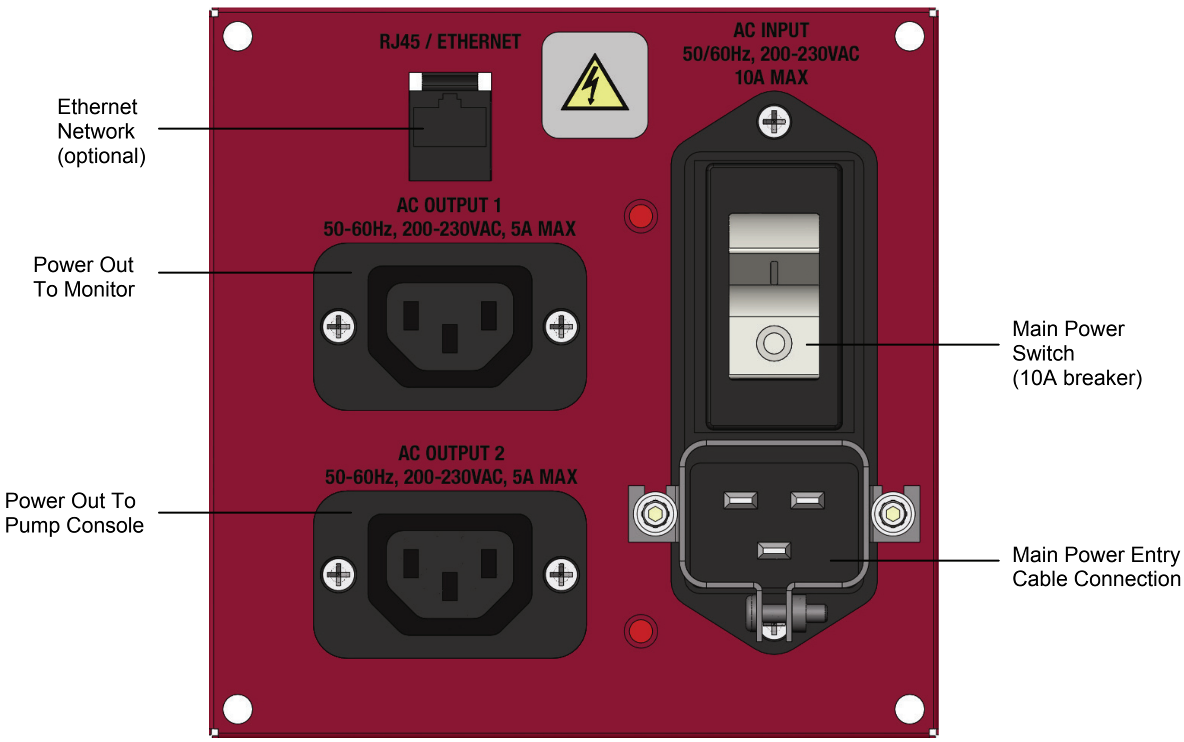


Figure 3-2. Power Distribution Unit

In addition to the main SQUID VSM cabinet, a pump cabinet houses a vacuum pump required for instrument operation. A printer for the computer sits on top of this cabinet. The power for this cabinet is provided by the main cabinet to prevent ground loops. A metal hose and a USB cable also connect this cabinet to the cryogenic insert in the main cabinet.

The instrument operation is most easily understood by recognizing several different subsystems that operate nearly autonomously to generate the desired instrument behavior. Many of these subsystems have components housed in different locations, such as in the pump cabinet, in the modular electronics bays, and in the cryogenic insert. All of the subsystems have hardware and software components, and their operation is coordinated by the MultiVu software on the computer.

These subsystems are:

- Temperature control system (section 3.3)
- Magnetic field control system (section 3.4)
- Motion control system (section 3.5)

- SQUID detection system (section 3.6)
- Chamber atmosphere control system (section 3.7)
- Cryogen monitoring system (section 3.8)

3.2.2 Modular Electronics

The electronic components of the MPMS SQUID VSM are housed in modular enclosures that are easily added to the system or replaced in case of upgrade or repair. (One exception is the gas handling controller that is tightly integrated on the top of the cryogenic insert.) These modular electronics communicate with one another and with the system computer using a proprietary, industrial-grade network based on the Controller Area Network (CAN) protocol.

The basic SQUID VSM electronics comprises:

1. a gas handling controller (integrated on the cryogenic insert, model EM-QD)
2. a magnet controller, model EM-QB (in the cabinet space above the computer)
3. a SQUID control module, model CM-F
4. a motor control module, model CM-A
5. a temperature control module, model CM-G

The SQUID control module, motor control module, and temperature control module are housed in module bays that provide power and CAN bus interfaces in a standardized format. Additional controllers supplied with upgrade options may also be housed in these bays.

W A R N I N G !

Always power module bay tower down before installing/removing modules.

Installing/Replacing Modules

The module bays are divided into two categories based on the heat that each must dissipate. The left-most bays are reserved for high-power modules, such as the motor control module and temperature control module. These modules will not fit in the other bays—do not force them or equipment damage may result. The center and right-most bays are for low-power modules, such as the SQUID control module. Low power modules may be placed in any bay. The excess cooling provided in high power bays will not harm them. You may distinguish low power modules from high power modules by the number of banana jacks on the rear panel: low power modules have two jacks, high power modules have one jack.

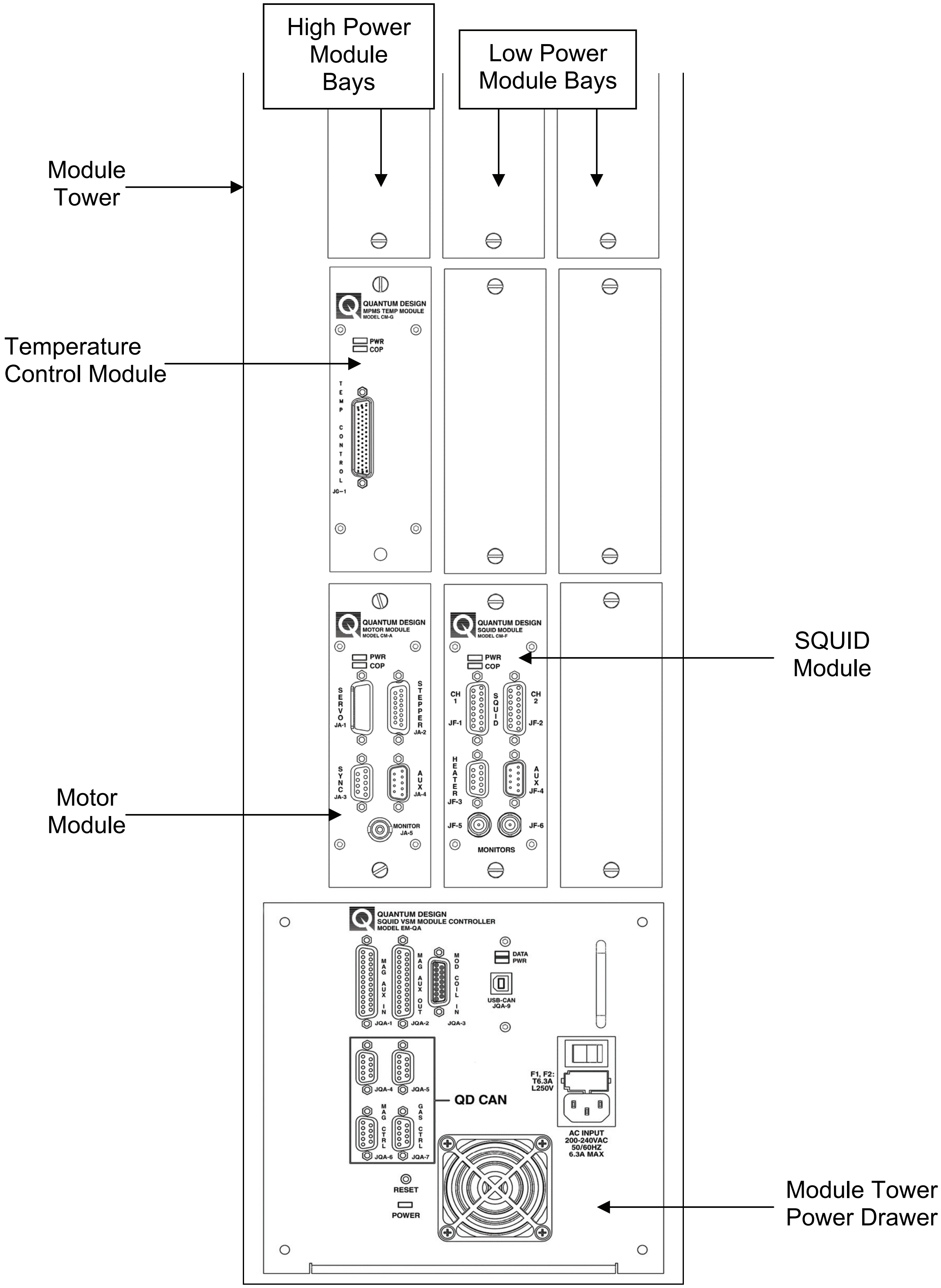


Figure 3-3. Module bay tower.

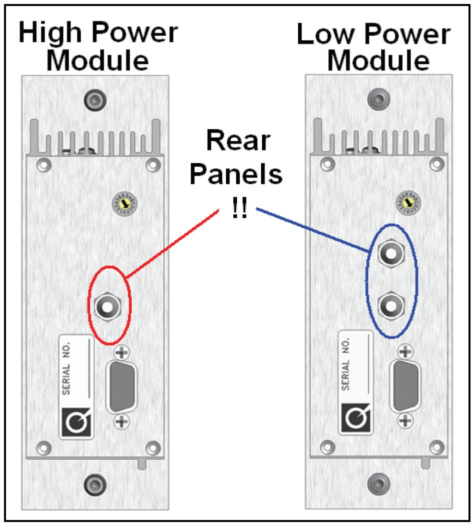
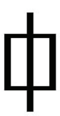


Figure 3-4. Module Rear Panels

The panel below the module bays provides access to a modular power supply. In addition, this panel has a connector for communication with the computer, a breakout connector for the auxiliary magnet signals (allowing access to the magnet's modulation coil by another device) and four connectors for connecting external CAN-based electronic controllers. The latter connectors provide +/- 24VDC fused power to external modules. The fuses are behind this panel and should only be replaced with equivalent 5A, 20 mm, delay fuses under the direction of a qualified Quantum Design service representative. It is possible for the fuse on a +24V line to open without affecting the fuse on a –24V line. In this case the electronics attached to these QD-CAN connectors may continue to operate with limited or unusual functionality. However, both fuses should always be replaced at the same time.



The model EM-QA module controller also utilizes two 6A, 20 mm delay fuses at the power entry. These should only be replaced with equivalent fuses on the direction of a Quantum Design service representative, and should always **both** be replaced at the same time.

3.2.3 Cryogenic Equipment

W A R N I N G !

Cyrogens such as liquid helium and liquid nitrogen pose the following dangers: (1) They can cause serious burns due to extreme cold. (2) They may expand explosively in closed containers if warmed above their boiling point. (3) Their vapors may displace the breathable air in closed room. (4) Liquid or vapors may backstream out of fill ports when removing caps/plugs/transferlines.

To guard against these dangers, wear protective clothing such as gloves and covered shoes when working with these cryogens. Also ensure that the room has good ventilation. Never disable relief valves, burst disks, or other safety devices on cryogen vessels and transfer equipment.

Experienced personnel should perform or supervise cryogen transfers.

Liquid helium and liquid nitrogen are required to operate the MPMS SQUID VSM. Tanks, tubes, and hoses for these cryogens are often insulated with an evacuated space around them and fitted with pressure relief mechanisms (such as burst disks and re-sealing relief valves) to prevent the buildup of dangerous pressures. For both performance and safety reasons, it is critical that no vacuum insulation nor pressure relief mechanism be tampered with, modified, or disabled. All users should also be aware of the burn and asphyxiation hazards posed by liquid cryogens. Please read section 1.5 for an overview of these hazards.

The purpose of the liquid helium is threefold. Liquid helium cools the superconducting solenoid (magnet) that provides the magnetic field in the instrument, and associated components of the magnet system such as two superconducting electrical leads and the quick switch. The use of a superconducting magnet system is the only practical way to generate magnetic field strengths found in the MPMS SQUID VSM. Liquid helium also cools the Superconducting Quantum Interference Device (SQUID) that is responsible for the instrument's extreme sensitivity to magnetic moments, as well as the SQUID's superconducting magnetic shield and the superconducting detection coils inductively coupled to the SQUID. By means of a vacuum pump, cold helium gas is drawn through the space surrounding the sample chamber to cool the sample down to 1.8K.

The liquid nitrogen in the instrument serves to minimize helium boil-off in several ways. First, the outside of the liquid helium tank is shielded from radiated heat by the liquid nitrogen tank. Second, the upper section of the cryogenic insert, which hangs into the helium tank, is connected to the liquid nitrogen tank to intercept conducted heat. Third, this connection is linked to a radiation shield inside the cryogenic insert. The radiation shield intercepts heat from the sample chamber and heaters which would otherwise radiate to the liquid helium from within.

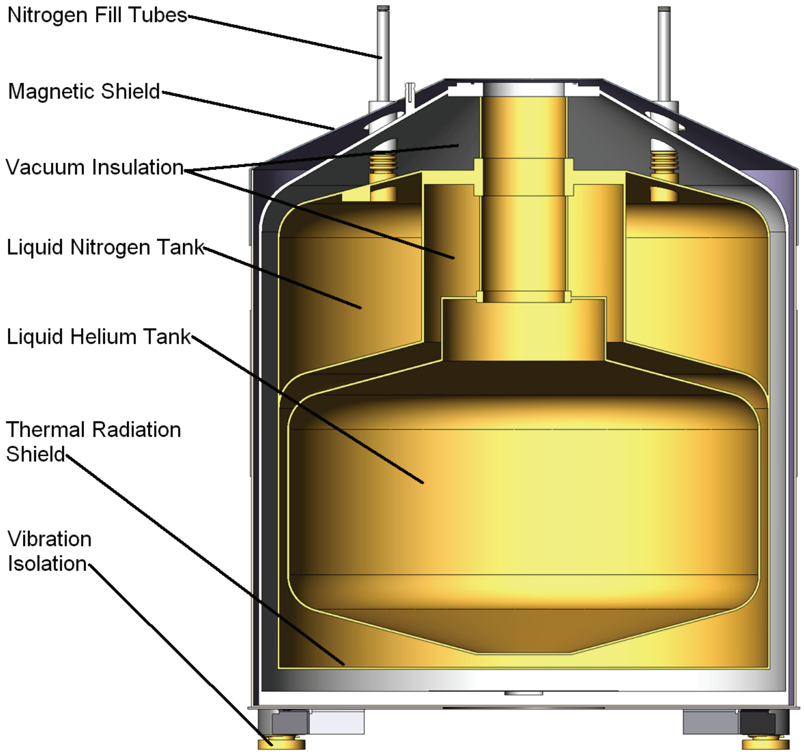


Figure 3-5. SQUID VSM Dewar

With the standard instrument cryostat, the hold time for the nitrogen tank when full, under typical operating conditions, is approximately 12 days. The hold time for the helium tank, when full, is slightly longer if there is liquid nitrogen in the nitrogen tank. When the liquid nitrogen tank is empty the instrument will **no longer achieve the lowest base temperature** specified. In addition, the rate of helium consumption will increase. When the liquid helium tank is empty, the SQUID detection system, magnet system, and temperature control system will all fail to work properly, and there is potential for damage to the hardware if operated with an empty helium tank. It is recommended to fill both the nitrogen and helium tanks at least every 10-12 days, or once a week if convenient. See chapter one for instructions to fill the nitrogen and helium tanks.

Except in EverCool systems, the helium and nitrogen that escapes from the cryogen tanks through normal evaporation is routed to hose barbs on the back of the main cabinet through 0.2 psi (1.4 kPa) relief valves. Rubber hose allow to route the exhausts to a visible location on the floor, to allow visual notification when the nitrogen tank is full during liquid nitrogen transfer. Liquid will be expelled from the hose when the nitrogen tank is full. The helium exhaust may be routed to a recovery system.

The N2 exhaust rubber hose features an inline one-way walve at about 80 cm (30 in) from the exhaust hose barb. This valve ensures that no air gets cryopumped into the N2 tank. It is recommended to check regularly that the hose section between valve and system is not collapsing while doing a HELIUM transfer. If this is the case, please contact QUANTUM DESIGN. There might be risk for plugging the N2 exhaust with ice.

3.3 Temperature Control System

The MPMS SQUID VSM sample chamber has a number of heaters and thermometers attached to the outside. The MPMS temperature module, model CM-G, monitors the thermometers and other data, reports the sample temperature, and automatically controls the power to the heaters to obtain the desired sample temperature. Cooling is achieved by drawing helium from the liquid helium tank into the annular space around the sample chamber at a controlled rate with a vacuum pump. The gas handling controller operates a valve, in line with a flow sensor, to achieve the necessary helium flow rate. To hold temperatures below the 4.2 K boiling point of liquid helium at one atmosphere, a capillary tube with carefully tuned flow rate allows liquid helium into the annular cooling region from the helium tank. The region is evacuated to low pressures while helium is continuously supplied. The evacuation is performed by a rotary-vane pump (except in EverCool systems) pumping through a solenoid valve that is used for isolating the region under special circumstances, such as power loss.

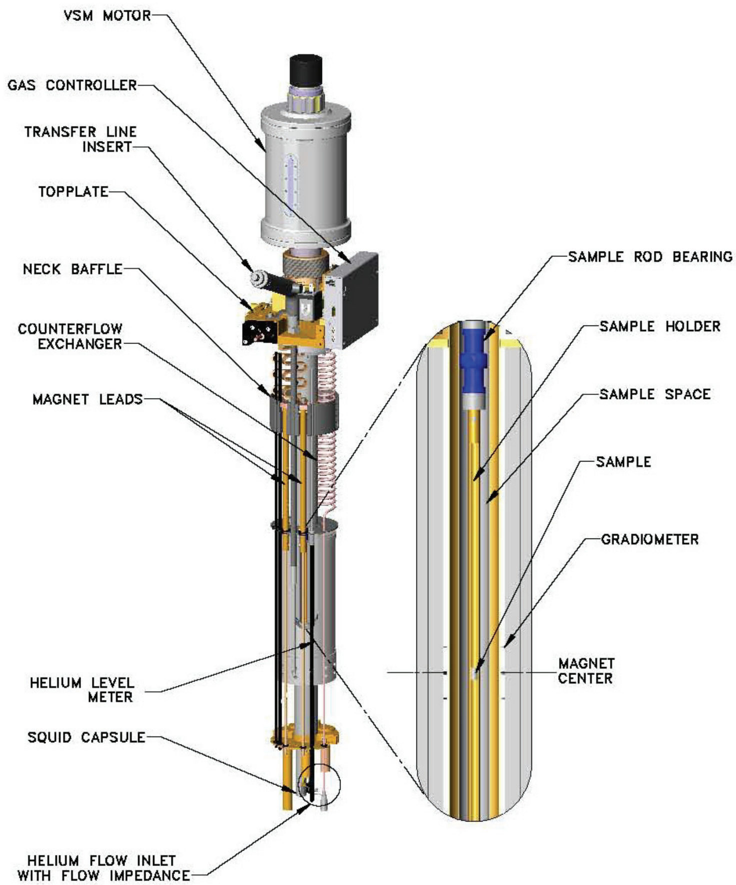


Figure 3.6. SQUID SVSM probe.

The reported sample temperature comes from a thermometer reading that has been corrected for a number of factors, including magnetic field, thermometer location, thermal history, and thermal conduction from gas flow past the sensor. These corrections can only provide an approximation of the exact sample temperature. The most accurate sample temperature data is achieved at steady state. Dynamic corrections, and the associated errors, are smallest when the rate of temperature change is small.

Occasionally, the flow impedance or the tube for drawing helium into the cryogenic insert may become plugged with ice or other solid contaminants. A plugged impedance will result in reduced helium flow through the probe insert and cause loss or degradation of temperature control below 10K. In this situation the flow reading of the ball gauges located on the side of the pump console, will generally be below 100 ccm. The only remedy is to warm the entire cryogenic insert to room temperature. This may take over a week to do if no accelerated warming techniques are used. It is recommended that only qualified Quantum Design representatives perform accelerated warming techniques on an instrument with this type of plug. To help avoid this condition, follow the tips below to keep contaminants out of the liquid helium tank and cryogenic insert.

- Use clean liquid helium, and do not pull liquid helium off of the very bottom of the storage dewar, where solid contaminants settle.
- Replace the cap on the helium fill port right away whenever transferring liquid helium into the helium tank. Never leave the tank open to atmosphere.

- Check the Teflon disk inside the cap of the helium fill port for dirt and debris before replacing it. Watch for poor sealing of the cap during normal operation and do not let a leaking condition persist.
- Be mindful of changes in the rate of helium evaporation coming out of the helium exhaust port on the back of the cabinet, if connected to a meter or monitoring system. An apparent reduction in helium evaporation rate might actually be caused by a leak between the tank and exhaust port which should be repaired.
- Never stress, stretch, kink, disconnect, or loosen the hose between the main cabinet and the pump cabinet when the instrument is in operation.

3.4 Magnetic Field Control System

The magnetic field in the MPMS SQUID VSM is generated by a superconducting Niobium-Titanium (NbTi) solenoid mounted on the outside of the cryogenic insert. The solenoid generates a vertical magnetic field. Current is supplied by the magnet controller via a set of permanent current leads. The leads are composed of different materials depending on what part of the system they are located in. In the cabinet, the magnet leads are large gage copper cable. In the very top section of the helium tank they are brass, to reduce the amount of heat that is conducted through them into the liquid nitrogen and helium. Below the brass section, where the temperature is low enough, the leads are composed of high-temperature superconductor (HTS) tape, to eliminate Joule heating. Below the HTS tape the leads are NbTi (type II superconductor) where they attach to the solenoid.

In parallel with the magnet solenoid is a superconducting element called the Quick Switch. The Quick Switch is immersed in liquid helium. It is driven above its critical temperature with a heater. In this normal state the magnet controller may generate a voltage across the superconducting solenoid. The solenoid current—and the magnetic field at its center—changes at a rate determined by the applied voltage and the solenoid inductance. The Quick Switch cools very rapidly and becomes superconducting when the heater is turned off. In this state there is a short across the solenoid and no voltage may be developed. Therefore, the magnetic field is extremely stable.

The magnetic field reported by the instrument is the current that is being driven through the magnet solenoid, multiplied by a geometric calibration factor. It is possible for the magnetic field lines within the instrument to move—a condition called “field relaxation.” It is also possible for a magnetic field to become trapped within the magnet even with no net current through the magnet solenoid. This condition, commonly called “trapped flux,” depends greatly on the magnet charging history. (See chapter 4 for more details.) A heater integrated into the magnet solenoid may be used to drive the wire above its critical temperature and eliminate most trapped magnetic flux. This procedure is called a *magnet reset.*

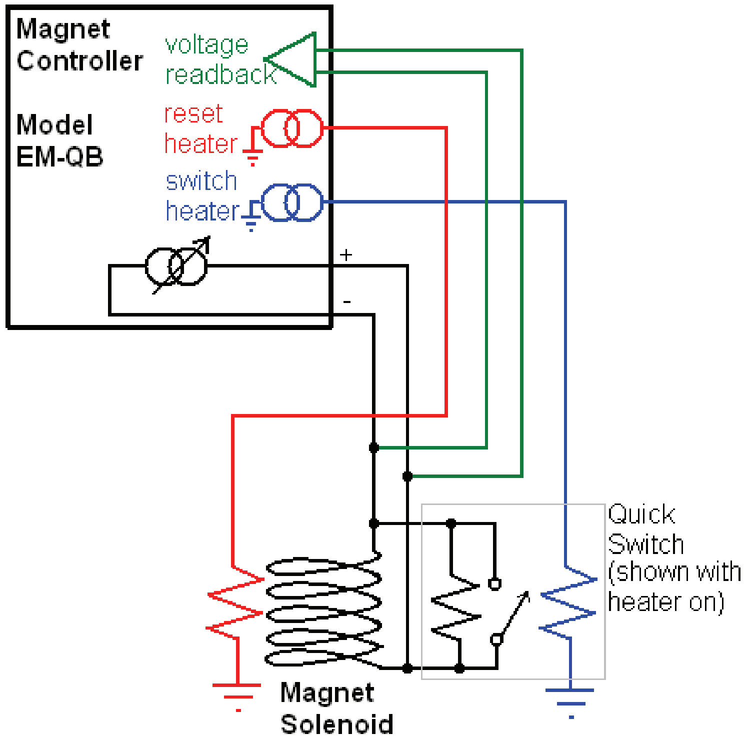


Figure 3-7. Magnet Control Diagram.

The magnet solenoid also contains a second, much smaller modulation solenoid for supplying small magnetic field offsets (to trim the magnetic field to true zero, low field option) and for generating AC magnetic fields (for AC magnetic susceptibility measurements, AC option).

The Magnet Controller, model EM-QB, coordinates operation of the Quick Switch and the current in the magnet solenoid. The novel design of this digital magnet controller utilizes analog feedback to achieve very smooth current ramps. Except at zero magnetic field, this controller always supplies current to the magnet solenoid and must not be disconnected from the magnet. Doing so could damage the magnet. This is different than the operation of many other superconducting magnet systems, which often have detachable or retractable leads.

The model EM-QB magnet controller utilizes two 8A, 20 mm delay fuses at the power entry. These should only be replaced with equivalent fuses on the direction of a Quantum Design service representative, and should always **both** be replaced at the same time. Spare fuses can be found in your utility kit.

3.5 Motion Control System

The motion control system moves the sample within the detection coils. The VSM head, or motor, is a long-throw linear motor mounted on springs within its casing for vibration isolation. The VSM head receives current from the motor control module, model CM-A. A DC signal controls the sample position, and an AC signal determines the vibration amplitude and frequency. A precision optical encoder in the VSM head reads the position of the motor armature to within 0.01 mm. This position is fed to the motor control module and used in a feedback loop to obtain precise sample positioning and vibration. The armature position may be seen through a window on the front of the VSM head.

The default vibration frequency is 14 Hz and has been selected to optimize sensitivity based on the mechanical properties of the instrument, but the vibration frequency is configurable as an advanced parameter. The vibration amplitude may be set below 0.1 mm, but 0.5 mm is the practical lower limit to achieve precise measurements. The maximum vibration amplitude allowed is 8 mm, but in practice will depend on the frequency used and the mass of the sample and sample holder. The default amplitude is 2mm. See the section below on the SQUID detection system for considerations when setting the vibration amplitude and frequency.

- The linear motor in the VSM head uses permanent magnets. **These magnets subject samples to approximately 200 Oe stray magnetic fields as they are inserted into the instrument**. Please contact Quantum Design if this is unacceptable for your samples.

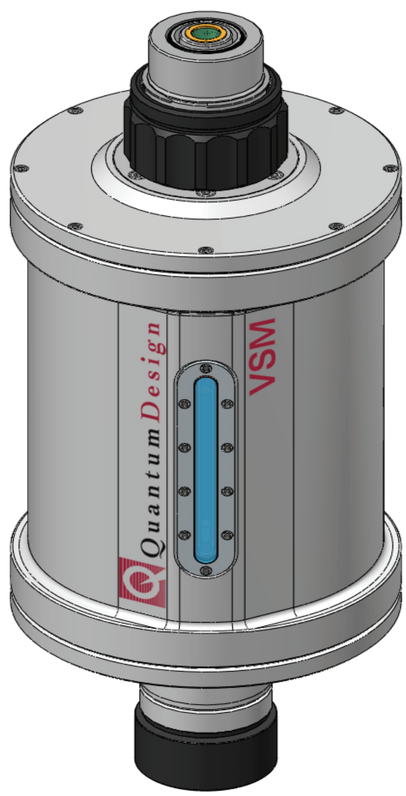


Figure 3-8. VSM Motor.

3.6 SQUID Detection System

CAUTION: SQUID detection is extremely sensitive to the magnetic signal generated by your sample, but also to external electromagnetic disturbances, such as for example cell-phones, rf-furnaces or stray-fields of equipment in close proximity. Environmental noise could affect the performances of your system. We advice to isolate your instrument from any known electromagnetic noise sources.

The SQUID detection system is composed of:

1. A set of superconducting detection coils inductively coupled to a magnetically-shielded, DC SQUID
2. A SQUID head that biases the SQUID, provides feedback control and signal amplification
3. A CAN-based SQUID control module with a digital signal processor (DSP) which performs synchronous AC detection

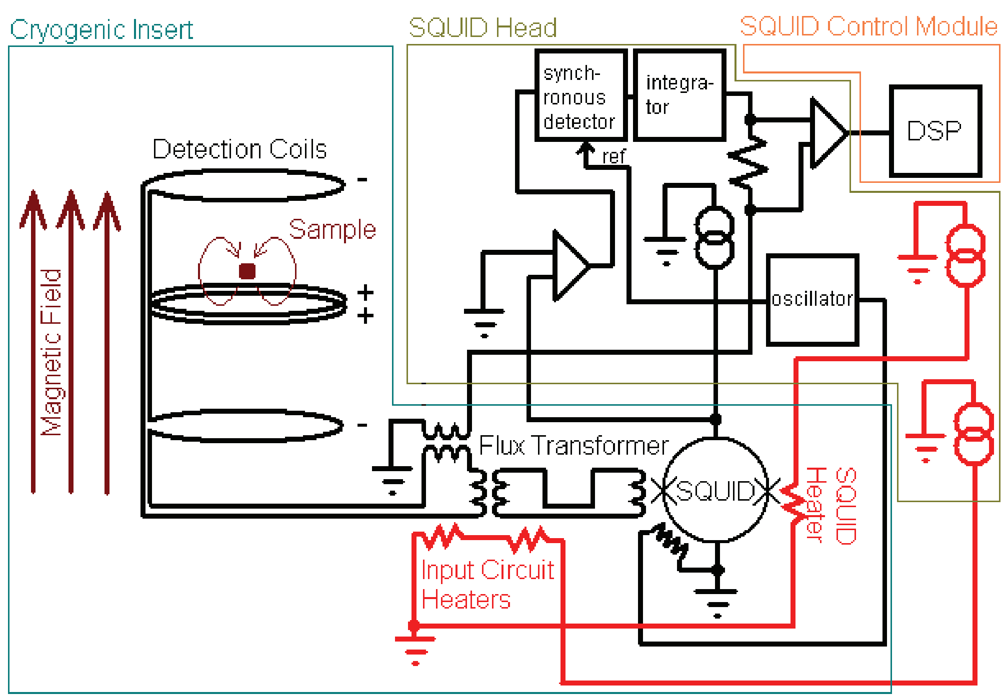


Figure 3-9. SQUID detection diagram.

The above diagram illustrates the detection system in simplified fashion. The detection coils are configured as a second order gradiometer to minimize signals from external disturbances. Both the detection coil circuit and the SQUID input circuit have heaters that allow the elimination of standing currents in the superconducting loops by raising them above their critical temperature, and which are operated automatically by the SQUID control module.

In the standard measurement, the instrument positions the sample at the center of the detection coil set, where the SQUID signal peaks. The sample is vibrated at 14 Hz and any measurable magnetic moment generates a 28 Hz SQUID signal at the input to the SQUID control module. A digital lock-in amplifier in the SQUID control module isolates this second harmonic (as well as the first harmonic) for analysis and data reporting. (See chapter 4 for more details on measurement theory).

- With this type of measurement it is extremely important that the sample be properly positioned at the center of the detection coils.
- If using a different vibration frequency, it is also important to select a frequency that is not too close to ½ the power line frequency, nor any harmonic thereof.

The SQUID ranges are labeled 1, 10, 100, and 1000. This corresponds roughly to the multiplier required to convert the output voltage from the SQUID electronics to the number of flux quanta (Φ_o) measured by the SQUID. Range 1 corresponds to the highest gain setting and is the *most* sensitive range. Range 1000 has the lowest gain and is the *least* sensitive range. When the sample produces a signal that is too large, the SQUID must be reset many times during the course of a single measurement due to repeated over-ranging as the sample vibrates. Little or no useful data is generated, or it takes much longer than the designated averaging time to generate a single data record. Using the autoranging function (default) minimizes the occurrence of such over-ranging.

- To decrease the sample signal with auto-ranging turned off and prevent such over-ranging, increase the SQUID range if it is not already set to 1000. Alternatively, reduce the vibration amplitude. The default vibration amplitude is 2 mm. The signal amplitude will decrease by the square of the reduction in vibration amplitude. (e.g. 1 mm vibration yields approximately ¼ of the signal size as 2 mm vibration.)
- Increase the vibration amplitude to generate a larger SQUID signal if the signal is too small to measure when the range is set to 1.
- The software estimates the maximum moment you may measure with given range and vibration amplitude settings. See the “Advanced” tab of the measurement dialog or sequence command pop-up.

3.7 Chamber Atmosphere Control System

During measurements, the sample chamber and VSM head are normally held in a medium vacuum—approximately 10 torr at room temperature. (The pressure may drop to 1 torr at the instrument’s base temperature.) Retaining a low pressure of gas in the chamber allows the sample and sample chamber walls to reach a uniform temperature. However, it is important that helium is the only gas in the chamber when cooling below room temperature. Water vapor, nitrogen, oxygen, and most other contaminants will condense and then freeze as the temperature is lowered, creating additional friction when the sample is vibrating, or even blocking sample motion altogether in extreme circumstances. Additionally, contaminants may impact some sensitive magnetic measurements, and may impact the instrument’s temperature control capabilities.

The standard procedure for inserting samples into the instrument involves purging the sample chamber and VSM head to ensure the clean environment described above. This procedure should be performed with the sample chamber at room temperature. The cap must be on the VSM head or the procedure will fail. The chamber is first evacuated by the system vacuum pump to a few torr, then it is filled with about one atmosphere of helium gas. (The helium vapor in the liquid helium tank is used, except in EverCool systems.) The chamber is evacuated and filled with clean helium gas in this manner twice more to flush out all contaminants, and then the chamber is evacuated to achieve the final pressure. Finally, the chamber is sealed.

When the VSM head is open for removing or inserting samples, clean helium gas is allowed to flow through the VSM head to prevent other contaminants from entering the head and chamber. This is called the "flooding" state.

- Avoid leaving the sample chamber open to atmosphere. Even though the chamber is automatically vented as a precaution to minimize sample chamber contamination, it is best practice to keep the cap on the VSM head whenever possible.

Besides the continuous venting state and the purging then sealing operation, the sample chamber may also be immediately sealed, or pumped continuously. All of these operations are performed with a series of solenoid valves on a manifold integrated onto the top of the cryogenic insert. The valves are operated by the CAN-based gas handling controller (Model EM-QD), also integrated onto the top of the insert.

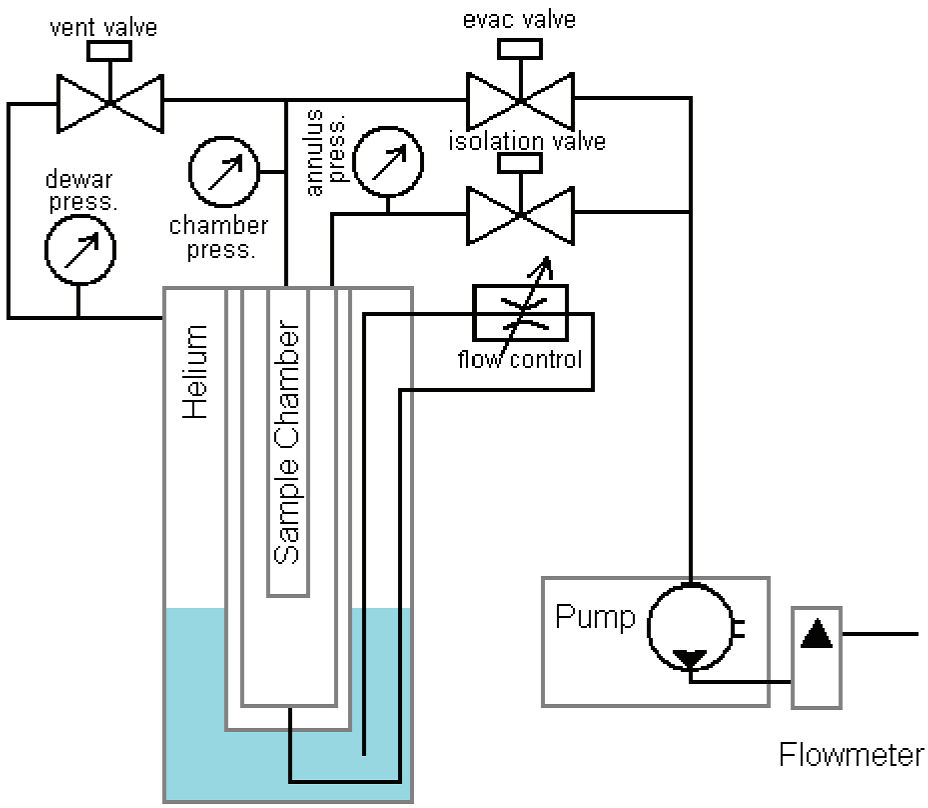


Figure 3-10. Gas Handling Diagram.

3.8 Cryogen Monitoring System

The liquid helium level is monitored with a superconducting wire with sufficient electrical current flowing so that only the portion of wire submerged in liquid helium is superconducting. The measured wire resistance is then proportional to the length of wire **not** immersed.

The helium level meter positioning and approximate calibration are shown below. The top of the meter is near the top of the magnet solenoid, and the bottom of the meter is near the SQUID and the inlet for the tube that draws helium into the cryogenic insert for sample cooling.

- It is not necessary to submerge the magnet solenoid in liquid helium. The magnet is cooled by helium vapor and by conduction through copper elements. In fact, the presence of bubbles in the liquid helium around the detection coils may increase noise in some sensitive measurements. Therefore, it is recommended to fill the tank to the bottom of the magnet solenoid, where the helium level reading is full (approximately 70 liters.) This much liquid helium may last 12-14 days under normal operating conditions, and if the liquid nitrogen tank is not allowed to run dry.
- The magnet being vapor-cooled, it is required to ramp the magnetic field to zero before starting a helium transfer. Otherwise, the warm gas introduced into the dewar might cause the magnet to quench.
- In case you wish to add additional liquid helium reserve, the software reports the approximate volume of liquid helium in the tank up to about 110 liters.

There is currently no meter in the MPMS SQUID VSM for monitoring the liquid nitrogen level. The rate of nitrogen evaporation will be fairly constant. The liquid nitrogen tank should be filled every 10-12 days. It is recommended to fill the nitrogen and helium tanks at the same time.

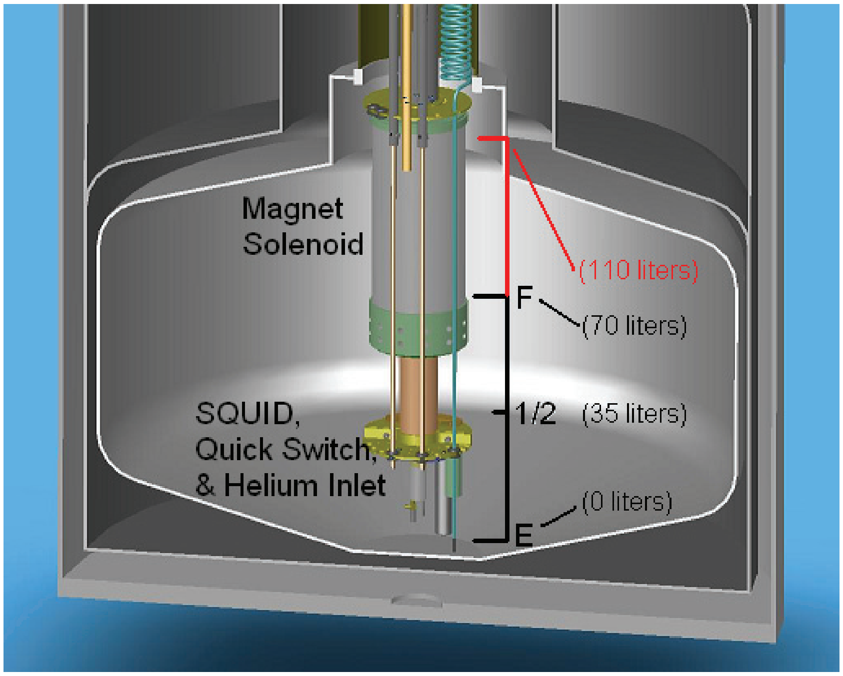


Figure 3-11. Helium level inside dewar.

CAUTION: Carefully read chapter 1 section 1.5 for detailed instructions on filling nitrogen and helium.

3.9 Additional Hardware

3.9.1 Sample Rods

The sample rods to which the sample holders attach are composed of tapered carbon fiber tubes, with adapters on either end. They are light, strong, and rigid—all important features for application in the SQUID VSM. A flexible plastic coupling at the top end helps accommodate slight misalignments between the magnetic latch mechanism and sample tube. The magnetic latch mechanism at the top secures the rod to the armature of the VSM head. A bearing at the bottom end of the sample rod provides a smooth surface to minimize friction against the walls of the sample chamber. The bearing also contains internal threads where sample holders attach.

- Store the sample rods in the storage space provided on either side of the lower half of the instrument cabinet to keep them clean and prevent damage. You must remove any attached sample holder first.
- Be aware of the strong magnets in the top of the sample rod as you move the sample rod around your laboratory. They may be attracted to other equipment or debris.
- Inspect the magnets at the top of the sample rod from time to time and make sure they are clean. Dirty magnets may prevent proper latching of the rod to the motor armature. The magnets may be cleaned with a cotton swab and mild detergent (no alcohol). Magnetized debris may be removed with lint-free tissue paper or adhesive-backed tape. If damaged, the magnetic latch may be replaced without replacing the entire sample rod. (See appendix C for ordering replacement parts.)
- Inspect the bearing at the bottom of the sample rod periodically, also, to be sure it is clean and not noticeably scratched, abraded, or worn. A dirty bearing should only be cleaned with a cloth and a small amount of isopropyl alcohol. Solvents such as acetone will damage this bearing.
- Leave the blue bearing on the rod. Verify periodically that the bearing is screwed all the way against the sample rod.
- When screwing the sample holder onto the sample rod, hold the blue bearing instead of the fragile carbon fiber rod to avoid damage. (see section 1.3.2)

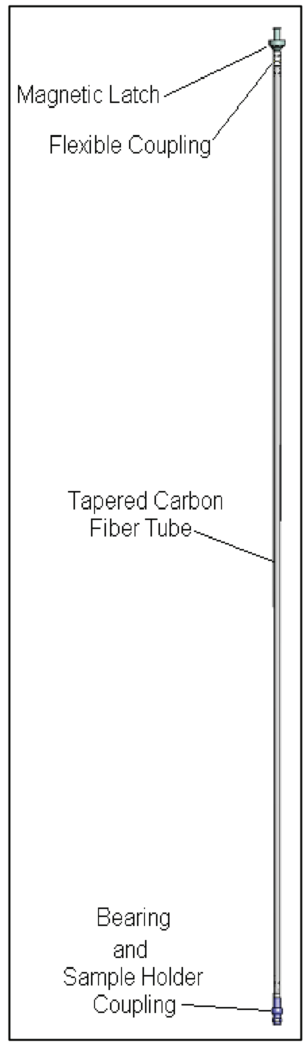


Figure 3-12. Sample Rod

3.9.2 User Kit

a. The user kit supplied with the instrument contains several items: rigid brass and quartz sample holders for mounting samples onto.

b. A sample mounting station to aid proper sample mounting as described in section 1.3.1.

c. A palladium sample reference sample, mounted to a sample holder, which may be used to check the instrument's magnetic moment calibrations with the formula below for the susceptibility of palladium at room temperature:

$$\mu=\chi_m Hm$$

where μ is the magnetic moment of the sample, χ_m is the mass susceptibility of palladium at 298 K (5.25×10^{-6} emu/Oe-g), H is the magnetic field applied by the instrument, and m is the mass of the palladium sample (supplied with the sample.)

d. A mounted Indium sample, which may be used to check the temperature accuracy of the system. The superconducting transition at zero field should be between 3.37K and 3.43K. Ensure that you have quenched all residual field out of the magnet when performing the M(T) measurement.

e. Two quartz sample braces for helping to secure samples inside the brass sample holder. These braces are intended to mount thin samples perpendicular to the magnetic field.

f. A set of powder sample holders, allowing to safely encapsulate powder samples and snap them into brass holders.

g. Spare parts for the sample rods: one flexure which mounts to the top of the sample rod and one blue bearing, which mounts to the bottom of the rod.

C H A P T E R 4

Measurement Theory and Applications

4.1 Overview

In this chapter we give detailed insight into the measurement theory and the most common applications. Section 4.2 outlines the VSM theory based on a 2ω detection principle and focuses on various critical effects which need considerations for using the system at its highest performance: sample geometry and sample holder effects, sample insertion and location effects, and vibration amplitude and frequency optimization. Section 4.3 provides advice on how to perform effective M(H), M(T) and M(t) sequences. Data file analysis tips and tricks are given in section 4.4, followed by example experiments (section 4.5) and a description of the most common sources of errors (section 4.6). Finally, we provide references for further reading (section 4.7).

4.2 VSM Measurement Theory

The MPMS SQUID VSM is a versatile and complex instrument. Optimizing your use of the instrument and obtaining the highest quality data sometimes requires deeper knowledge than the fundamentals of sample insertion and software operation. The sections below help explain the practical use of the instrument in detail, with specific examples of common magnetic measurements and numerous important experimental considerations.

The illustration below is a simplified model of the MPMS SQUID VSM detection hardware. The superconducting detection coils are configured as a second-order gradiometer, with counterwound outer loops which make the set of coils non-responsive to uniform magnetic fields and linear magnetic field gradients. The detection coils only generate a current in response to local magnetic field disturbances.

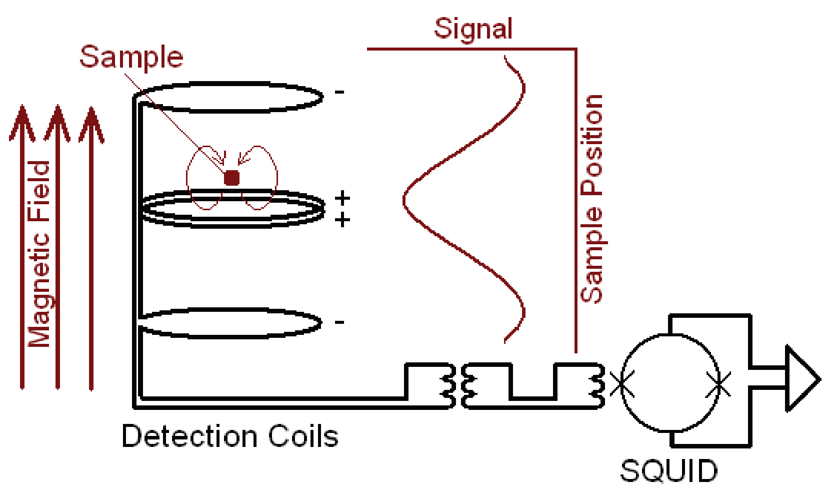


Figure 4-1. SQUID detection schematic.

Assuming the sample dimensions are much smaller than the dimensions of the detection coils, the current in the detection coils is a function of the sample position. The approximate shape of this function is shown above. The exact shape varies slightly with the size and shape of the sample. The current in the detection coils is inductively coupled to the instrument's SQUID, which serves as an extremely sensitive current-to-voltage converter. Many details of the SQUID circuit are omitted for simplicity here, but it is relevant to note that SQUID feedback nulls the current in the detection coils so no current actually flows in them, and the feedback current yields the actual SQUID voltage for analysis. See the "Further Reading" section at the end of the chapter for references that explain the details of SQUID operation. The SQUID voltage is amplified and digitized by the instrument electronics.

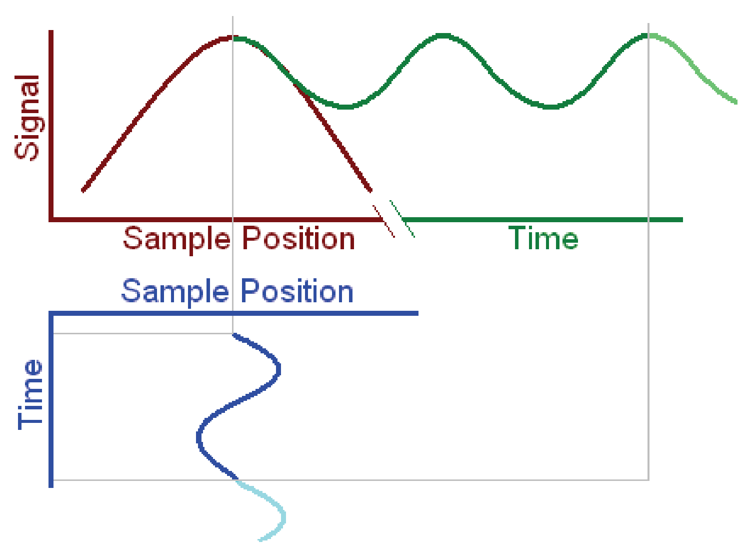


Figure 4-2. 2ω detection principle.

The SQUID VSM measurement technique vibrates the sample at frequency ω about the very center of the detection coils, where the signal peaks as a function of sample position, z. This generates a SQUID signal, V, as a function of time, t:

(1) $$V(t) = AB^2\sin^2(\omega t)$$

because $V(z) = Az^2$ for small vibration amplitudes, and $z(t) = B\sin(\omega t)$. Here, A is a scaling factor relating to the magnetic moment of the sample. B is the amplitude of sample vibration. Since $\sin^2(\omega t) = ½ - ½ \cos(2\omega t)$ (by identity) the techniques of a lock-in amplifier may be applied to isolate and quantify the signal occurring at frequency 2ω, which should be caused exclusively by the sample if the vibration frequency is selected wisely. Briefly, this is achieved by multiplying the measured signal with a phase-corrected reference signal at 2ω and then extracting the DC component of the result. This DC component is proportional to the 2ω component of the measured signal.

This technique quickly and precisely isolates the sample signal from other noise sources, including drifting SQUID signal and mechanical noise sources synchronized to the sample vibration. The lock-in amplification of the SQUID signal is performed by digital electronics in the SQUID control module.

4.2.1 Sample Geometry Effects

4.2.1.1 SCANNING FOR SAMPLE OFFSET

When scanning the sample to determine the sample offset, the software measures the 2ω signal component for various vertical sample positions, as described in the measurement theory section, then calculates and displays a fit to this data. The fit function is derived from a point dipole in a uniform magnetic field and is therefore *not* identical to the shape of the function for the palladium reference nor any other real sample, but is very similar and will accurately locate small, uniform samples. It is also not identical to the spatial response curve plotted in the measurement theory section above, which is a plot of the *DC* response of the detection coils. It is similar to the second derivative of the DC spatial response curve.

If the scan data shows clear deviations from the fit function, this may demonstrate the sample's deviation from point-like dipole behavior due to its size shape, or magnetic characteristics. Alternatively, it may be an effect of a non-uniform magnetic field. For example, a non-uniform residual magnetic field after the magnet is set to zero field is common. In either case, you should carefully consider whether the calculated sample offset is accurate or not.

4.2.1.2 DETECTION COIL GEOMETRY

For reference, the dimensions of the MPMS SQUID VSM detection coils are 17 mm (diameter) and 8 mm (height, center to either outer coil).

The instrument is calibrated with a right circular cylinder of palladium that is supplied with the instrument, with diameter 2.8 millimeters and height 3.8 millimeters. To the extent that your sample varies from this geometry, the instrument readings will be impacted in the following ways:

4.2.1.3 LENGTH EFFECTS (LONG/SHORT SAMPLES)

For samples significantly longer than the palladium reference, the material at either end of the sample generates more magnetic flux for the outer, counterwound detection coils to pick up. This has the effect of flattening the spatial response curve of the detection coils—reducing the magnitude of A in equation 1. The result is that the reported magnetic moment appears smaller than the actual magnetic moment. A very long, uniform sample would not register any signal at all as long as both ends are far away. Recommended sample length is < 5 mm.

Similarly, for samples significantly shorter than the palladium reference the spatial response curve is taller and the reported magnetic moment appears to be too large. Since the magnitude of A in equation 1 is determined empirically with a reference sample, one may counter this effect by carefully measuring a reference of known magnetic moment and with geometry similar to that of the unknown sample, and then normalizing all magnetic moment measurements for the unknown sample to this well-known measurement.

4.2.1.4 RADIUS EFFECTS (WIDE/NARROW SAMPLES)

The lines of magnetic flux due to a sample will, on one scale or another, look like those from a magnetic dipole. That is, they emanate from the sample and return to the sample. Because the sample cannot completely fill the detection coils, some of these magnetic flux lines return inside the detection coils and are therefore not detected. This is true for all samples and the magnitude of this effect depends on the radius of the sample relative to its length and relative to the radius of the detection coils.

Samples narrower than the calibration reference will usually generate smaller signals than expected and samples wider than the calibration reference will usually generate larger signals than expected. In other words, if you reduce the radius of a palladium reference sample by a factor of 2, its volume decreases by a factor of 4: the instrument will report a reading more than a factor of 4 smaller even though the material has the exact same volume magnetization. The magnitude of this error is described by the term *filling factor*.

This effect can also be largely accounted for by carefully measuring a reference of known magnetic moment and with geometry similar to that of the unknown sample, and then normalizing all magnetic moment measurements for the unknown sample to this well-known measurement.

4.2.1.5 DEMAGNETIZATION FACTOR

Samples with a large magnetic susceptibility and certain geometry effectively alter their own internal magnetic field. Accounting for this magnetic field correction can be an important consideration for many magnetic measurements. The demagnetization factor is a geometry-based value between zero and one that describes the extent of this field alteration. This effect is due only to the sample geometry and susceptibility. It is not an instrumental artifact. The effect is especially relevant for wide, thin samples oriented perpendicular to the magnetic field—thin films in the extreme case—but should be considered for most samples. For example, the demagnetization factor in a spherical sample is 1/3, which can be significant if the sample has a large susceptibility. It is beyond the scope of this manual to derive or tabulate demagnetization factors for different sample geometries, but they may be located fairly easily in many textbooks and online references.

4.2.2 Sample Holder Considerations

4.2.2.1 CHARACTERIZING SAMPLE HOLDER CONTRIBUTIONS

Measure the magnetic signal from your sample holder *without* a sample mounted to it in order to characterize its contribution to your measurements.

4.2.2.2 IMPURITIES

Impurities in a sample holder may generate signals in the instrument. If a sample holder is not uniform in composition, but contains localized impurities, each point of impurity generates a local disturbance in the magnetic field which the detection coils will detect.

4.2.2.3 SAMPLE MOUNTING

The sample must be mounted *securely* to the sample holder. The sample holder must be connected securely to the sample rod. If the sample does not oscillate synchronously with the VSM head encoder array, then the instrument rejects much of the sample signal as out-of-phase. Quantum Design does not supply material for securing the sample to the holder. There are many glues and tapes available, depending on the specific application. Most have some magnetic contamination, especially at very low temperatures, so be sure to measure the glue or tape you use to determine the extent of signal it produces.

4.2.2.4 END EFFECTS

In an idealized measurement, the sample holder is a long, uniform material. In this case, the sample holder produces no net signal when it is inside the detection coils, because any magnetic field disturbance it produces is produced in the same magnitude at the two counterwound outer detection coils and the central coil. If a sample holder does not pass completely through the detection coil set, then it creates more local magnetic field disturbance at the top coil than at the bottom coil, and there is a net signal due to the sample holder. This is commonly referred to as a sample holder *end effect*. To prevent this end effect from impacting measurements, the sample should be mounted at least 50 mm from *both* ends of the sample holder, and should be the same distance from both ends. Also, any non-uniformities in the sample holder—such as bearings, fittings, and adapters—should be duplicated symmetrically about the center of the sample holder.

4.2.2.5 SUPPLIED SAMPLE HOLDERS

The sample holders supplied with the instrument are approximately 130 mm long, allowing the sample to be mounted 66 mm from the lower end. The VSM head will operate in the center of its position range. The quartz sample holders are the best to obtain the highest instrument sensitivity and accuracy. The quartz holders are brittle and fragile. For applications where the highest sensitivity and accuracy are not required, the brass sample holders are more robust and easier to handle.

4.2.3 Sample Insertion Considerations

4.2.3.1 INSPECTION

Each time you insert a sample into the instrument, inspect the following items to ensure optimal performance:

- The magnetic lock should be clean.
- The flex joint just below the magnetic lock must not be cracked or damaged.
- The coupling of the flex joint to the magnetic lock must be secure.
- The coupling of the sample holder to the sample rod must be secure.
- The sample holder must be mated straight for proper radial centering within the detection coils.
- The sample holder and your sample should be clean.

4.2.3.2 SAMPLE EXPOSURE TO MAGNETIC FIELDS

As you insert the sample into the instrument, the sample is exposed to a magnetic field of approximately 200 Oe inside the VSM head. A small remanent field is also normal inside the sample chamber. Oscillate mode will yield the lowest remenant magnetic field.

4.2.4 Sample Location Considerations

4.2.4.1 AXIAL LOCATION ERROR

It is very important to properly locate the sample axially when performing VSM measurements in the MPMS SQUID VSM. The graph below shows the error in the reported measurement of a palladium reference sample as a function of axial position offset for a 2mm vibration amplitude. Use this to help estimate the uncertainty in your magnetic moment readings based on your uncertainty in the sample position.

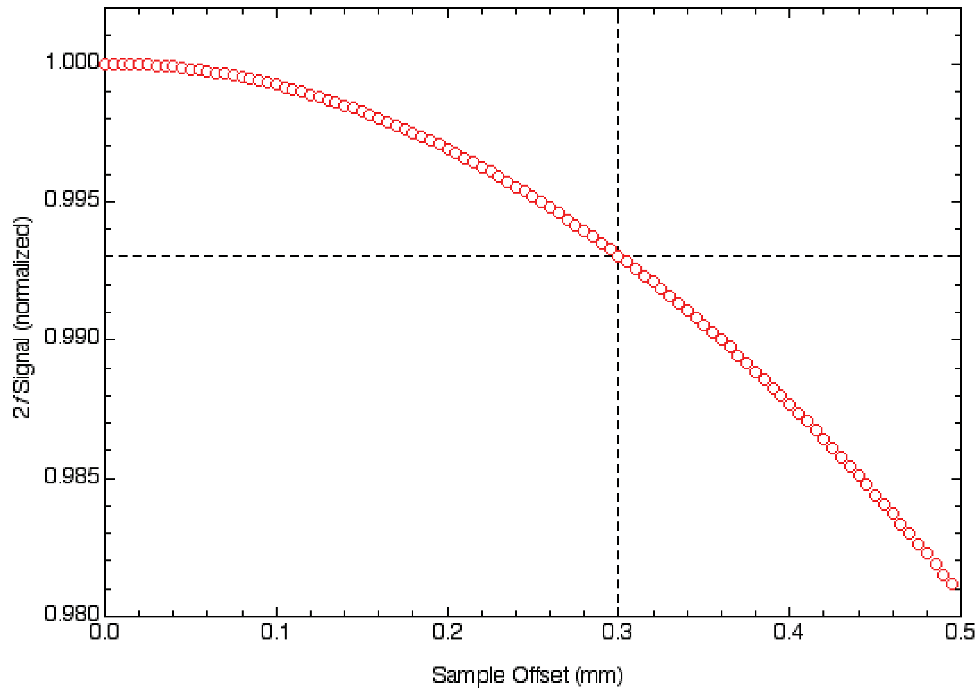


Figure 4-3. Sample offset accuracy.

- **Accuracy of sample offset scan:** When the sample has a moderate to large magnetic signal (> $1x10^{-4}$ emu) and produces a clean, symmetric signal for analysis, the sample position determined by the scan is typically precise to $1x10^{-5}$ m (10 µm). When the sample signal is weaker or the scan produces an asymmetric signal, the sample position determined by the scan may be precise to $5x10^{-5}$ m (50 µm), but in this case you should perform repeated scans to verify the reproducibility yourself. For very weak signals, large sample holder effects, and cases with magnetic contamination, the scan may actually produce meaningless data.
- **Accuracy of auto-tracking function:** The auto-tracking algorithm used to adjust the sample location based on the type of sample holder in use and the thermal history of the sample chamber has been demonstrated to accurately predict the sample location to within $5x10^{-5}$ m (50 µm). The auto-tracking function may only be accurately applied to the brass and quartz sample holders.

4.2.4.2 ANGULAR DEPENDENCE

It is also important to mount the sample so that it vibrates on the axis of the sample rod. If the sample vibrates off-axis, you may find that the magnetic moment readings depend on the angular orientation of the sample holder and sample rod when inserted, to a degree that is extremely difficult to quantify. This is because the instrument's geometry diverges slightly from the ideal geometry: detection coils have lead-in wires and bend with non-zero radius, the sample chamber may not be perfectly co-axial with the detection coils, etc. Rotate the sample rod to find the minimum moment being reported.

4.2.4.3 MAGNETIC IMPURITIES

Magnetic impurities in the sample holder, glue, or securing agent can generate erroneous signals and errors in sample offset scans. Measure the empty sample holder to verify that it is free of magnetic impurities. Routinely measure your glue or securing agent to verify that it is effectively free of magnetic contamination.

4.2.5 Vibration Amplitude Effects

4.2.5.1 SCALE THE SIGNAL BY CHANGING THE VIBRATION AMPLITUDE

The sample signal scales quadratically with the vibration amplitude. Therefore, changing the vibration amplitude is an effective way to scale the signal when it is too large or too small. Increase the vibration amplitude if the sample does not generate a large enough signal. Decrease the vibration amplitude if the sample generates too large a signal. The instrument accounts for this signal dependence so the reported moment remains accurate when you change the vibration amplitude. Because the vibration amplitude range of the instrument is effectively one order of magnitude (0.5 mm – 8.0 mm), this technique yields two orders of magnitude in the instrument's dynamic range for magnetic measurements.

4.2.6 Vibration Frequency Effects

4.2.6.1 DIFFERENCE FROM CONVENTIONAL VSM(S)

Unlike conventional copper-detection-coil vibrating sample magnetometers, a changing magnetic flux is not strictly required to generate a signal in the MPMS SQUID VSM. The sample vibration is used only to create a signal at a known modulation frequency and aid the separation of the sample signal from instrumental artifacts, not to generate the signal itself. The size of the signal does not depend on the vibration frequency and higher vibration frequencies will not improve the signal-to-noise ratio. This is due to the use of superconducting detection coils which produce a current in response to magnetic flux, rather than to a *change* in magnetic flux as produced by copper coils.

4.2.6.2 CHANGING THE VIBRATION FREQUENCY

The default vibration frequency corresponds to a mechanically quiet frequency for the system as a whole. There is little reason to use any different vibration frequency. In fact, by using a different frequency you may inadvertently detect additional noise due to electromagnetic or mechanical resonances in the instrument or within your laboratory. If your laboratory contains a source of noise, you may find it useful to change the vibration frequency of the MPMS SQUID VSM. Remember that the main signal of interest occurs at twice the vibration frequency. Therefore, it is wise to avoid vibration frequencies that are ½ of the power line frequency in your laboratory, and all higher harmonics. The AC power in the laboratory (usually 50 Hz or 60 Hz) is by far the most predominant source of erroneous signal for instruments such as the MPMS SQUID VSM. When using a different vibration frequency it is recommended to verify measurement accuracy using the supplied palladium reference. To determine the best frequency to use, you may wish to connect a spectrum analyzer to the BNC output (JF-5) on the front of the SQUID control module, model CM-F. Examine the frequency spectrum with no sample installed.

4.2.7 Averaging Time

The instrument sensitivity improves as the inverse square root of the averaging time. In theory, $2x10^{-8}$ emu moment may be resolved with one second averaging time, while $2x10^{-9}$ emu moment may be resolved with 100 second averaging time. Accordingly, one would expect $6x10^{-8}$ emu sensitivity with 0.1 second averaging. However, averaging time less than about 0.25 seconds is not recommended due to limitations of the control computer. For very long averaging times, instrument sensitivity will ultimately be limited by 1/f noise sources. Environmental noise sources may become significant.

4.3 Sequence Considerations

4.3.1 Moment Versus Field (M(H))

The SQUID VSM is designed to perform efficient M(H) loops. The most accurate data is measured when the instrument stabilizes the field for each measurement, which happens very quickly. It is also possible to collect data continuously while changing the magnetic field. The instrument noise floor will increase to over 10^{-6} emu if you measure while ramping. In addition, since the magnetic field and measured moment actually change over the course of such a measurement, the reported values will be averages, which tends to result in "blurry" or imprecise data.

Another consideration for some M(H) measurements is magnetic field error due to remanent magnetic field. The remanent field depends on the recent history of the magnet and is neither known nor reported. For more about remanent magnetic field, see the section below called "Common Sources of Error: Magnetic Field Error."

4.3.2 Moment Versus Temperature (M(T))

The SQUID VSM is designed to perform large temperature changes quickly and to quickly stabilize the temperature. It is also designed to maintain user-defined temperature sweep rates and to perform efficient M(T) measurements. A high data density may be achieved by measuring while changing the temperature. It may prove more efficient to sweep the temperature at a moderate or slow rate and collect data continuously, rather than stabilize the temperature for many measurements. To verify the accuracy of the reported sample temperature while sweeping temperature, occasionally stabilize the temperature and continue measuring. The reported temperature and magnetic moment readings will be averages, which can result in "blurry" or imprecise data, depending on the rate of temperature and moment change.

4.3.3 Moment Versus Time (M(t))

The speed of the VSM measurement technique makes it feasible to examine some time-dependent magnetic effects with the MPMS SQUID VSM. However, the time required to stabilize the magnetic field with the quick switch is on the order of 1 second, and the minimum time required to collect one data point (minimum practical averaging time) is about 0.10-0.25 sec, depending on the computer workload. Time-dependent effects faster than this cannot be resolved with the standard SQUID VSM measurements.

To view time-dependent effects, collect a continuous stream of data. You will need to carefully select the measurement averaging time based on the time resolution and moment resolution required. To examine a sample's remanent magnetization as a function of time, set the instrument magnetic field to zero using the fastest charging rate. If the instrument remanent magnetic field drifts after stabilizing at "zero" field, you may try to stabilize the magnet with the "Utilities > Reset Magnet" command. See "Common Sources of Error: Magnetic Field Error" below for more about remanent magnetic field.

4.4 Data File Analysis

4.4.1 Data Recorded in the Data File

Each SQUID VSM data record consists of a large number of data items. In addition to expected items such as magnetic moment, moment standard error, temperature, magnetic field, and time stamp, each data record contains information such as the averaging time, vibration frequency and amplitude, center position, SQUID range, in-phase and quadrature (out-of-phase) signal component in volts, minimum and maximum temperature and field during the course of the measurement, and hardware status codes. This large amount of information aids data analysis and troubleshooting.

4.4.2 Handling Large Data Files

The automation and rapid data collection capabilities of the instrument allow you to generate very large amounts of data, which can be difficult to manage in spreadsheet programs. The export utility described in chapter 2 helps you extract portions of the data from a data file. Alternatively, you may collect only a fraction of the data or set longer averaging times to reduce the number of data points collected.

4.4.3 Verifying Measurement Precision

To determine the measurement precision, plot the moment and the moment standard error ("M Std. Err.") against the measurement time, or other independent variable. Compare the standard error to the moment reading to determine the measurement uncertainty as a percentage of the total reading. Also, compare the quadrature signal ("M. Quad. Signal") to the reported moment—this value must also be small for precise measurements. The quadrature (out-of-phase) signal indicates the quality of the vibration-phase-sensitive detection. The standard error indicates the precision of the in-phase signal component.

4.5 Example Experiments

Detailed descriptions of how to perform several common experiements with the MPMS SQUID VSM are found in the sections below. It is assumed you are already familiar with the common tasks outlined in chapter 1 such as mounting and inserting samples, and have read all the application considerations described above.

4.5.1 Five Quadrant Hysteresis Loops

Hysteresis loops are easily performed using the "Moment vs. Field" sequence measurement command. Start by writing the following sequence, using the temperature and field parameters best suited for your research:

Set Temperature…

Wait For Temperature, Delay 10 secs…

SQUID VSM Moment vs Field…

Set Field 0.0 at 500 Oe/sec, oscillate, stable

End Sequence

The first line sets the temperature at which you want to perform the hysteresis loop, and the second line waits for 10 seconds after that temperature is achieved (for stability) before allowing the hysteresis loop to begin. The end of the sequence returns the field to zero in order to power down the magnet power supply and prepare for removing the sample. While not strictly required, it is good practice to keep the magnetic field at zero when no field is necessary in the instrument.

For the moment vs. field command, you must choose a large number of measurement parameters:

- Select and drag the field ramp shape in the "Select Start/End Quadrant" area to define the starting field and applied maximum and minum fields. A five-quadrant hysteresis loop would normally begin at $H_0 = 0$ and ramp up to H_{max}, then down to H_{min}, and back to H_{max} again as shown. The illustration shows a 2 Tesla, 5-quadrant hysteresis loop.

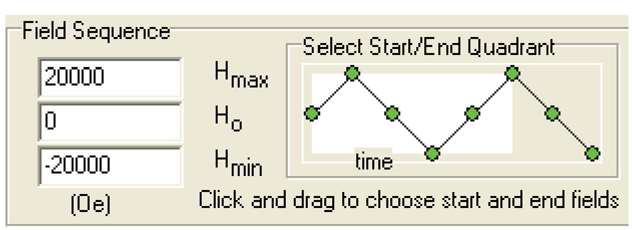


Figure 4-4. Hysteresis loop setup.

- In the “Field Control” area, select the magnetic field charging rate (“sweep” rate) and select whether to stabilize the field at each measurement field or to continue charging (“sweeping”) as measurements are performed. This decision will depend on your sample and on your research demands. Stabilizing at each field will yield greater measurement precision and accuracy, but *may* require more time. Each data record will report the **average** magnetic field and **average** magnetic moment obtained over the course of the measurement, so long averaging times and fast sweep rates can have a blurring effect on the hysteresis loop data if you do not stabilize the field for each measurement. You may reduce this systematic blurring effect by reducing the measurement averaging time (see below) and by reducing the field sweep rate. Data records that are being collected while a range change occurs are discarded, so measuring while sweeping could result in occasional holes in the hysteresis loop data, since the magnet charging does not stop to re-acquire the discarded record. If sweep continuously is not selected, the instrument will try to obtain the desired number of measurements before charging to the next field by re-acquiring data that was discarded due to a range change.

- In the “Data Acquisition” area, select the data spacing. The most common spacing for 5-quadrant hysteresis loops are probably “uniform spacing in field” and “continuous measuring.” With continuous measuring you specify the averaging time and the measurements are performed in rapid succession. This may not be particularly useful if your ramp will stabilize at each field—it is intended to be used with the “sweep continuously” mode selected. (A setting on the advanced tab allows you to use “sweep continuously” as a prerequisite for “continuous measuring.”) Since the continuous measuring selection can generate large amounts of data, you may also choose to keep only a fraction of the data records, and discard the rest. With uniform spacing in field all records are kept. With uniform spacing you also specify the number of fields between H_{min} and H_{max} at which to initiate measurement, or specify the field increment between measurement initiation. Finally, you may specify a number of times to repeat measurements at each field. (This is not meaningful for continuous measuring.)

- Examine the “Approximate Fields” box and “Estimated” box. This will show you the approximate fields where measurements will be initiated, and the estimated time to perform all the measurements. Make sure these estimates both meet with your expectations for the hysteresis loop you wish to perform.

- If you know that you need to adjust the vibration amplitude or frequency, the SQUID range setting, the automatic centering setting, or the magnetic field approach mode, or if you need to introduce a wait time prior to collecting each data record, you may control these features on the “Advanced” tab of the moment vs. field measurement window. For most applications, the default settings will work best.

Next, save your sequence. To perform the hysteresis loop measurement, perform the following steps:

1. Mount your sample to a sample holder and attach it to a sample rod.
2. Insert your sample into the instrument and place the cap on the instrument.
3. Specify the data file you want the data recorded to. (This is part of the sample installation wizard.)
4. Purge and seal the sample chamber. (This is also part of the sample installation wizard.)
5. Select and run your sequence.

6. On the control center, click the "view" button to plot your data as it is being collected. Recall that there might be a wait period while the instrument achieves your desired temperature before collecting data. To see the data in standard M vs. H hysteresis loop format:

    a. Right-click the data file window and select "Data Selection."
    b. For X-axis, select magnetic field, and for Y1-axis, select moment.
    c. For Y-2 axis, select moment standard error, to also display the uncertainty in each data record.
    d. Check "auto" for each axis to automatically scale the plots to show all data records.
    e. Click "OK."

4.5.2 Zero Field Cooling

Cooling your sample in truly zero magnetic field, as desired for characterizing some superconductors, requires careful consideration when inserting your sample into the instrument. Since the magnet solenoid windings tend to trap magnetic flux, it is common for the instrument to report zero magnetic field even though there is still a small field in the chamber. (See the section above, "Sample Insertion Considerations" as well as the section below, "Common Sources of Error: Magnetic Field Errors.")

To minimize the trapped magnetic flux, use the "oscillate" approach mode when setting the magnet to zero field. The command "Utilities > Reset Magnet" may also be used to eliminate trapped magnetic flux by driving the windings above their critical temperature.

A zero field cooling experiment might begin, for example, with the following steps:

1. Mount and insert a standard paramagnetic reference, such as Dy_2O_3
2. Set the instrument temperature so your reference has high susceptibility (20 K for Dy_2O_3)
3. Set the magnetic field to 10,000 Oe (linear mode), wait for stable field and temperature, then measure sample.
4. Determine susceptibility of the reference: moment divided by field (m/H).
5. Set the field to 50,000 Oe (linear mode), wait for instrument to reach this field.
6. Set the field to zero *in oscillate mode*, wait for instrument to reach zero field.
7. Use "Utilities > Reset Magnet" to minimize trapped flux in the magnet. Click "Yes" when the confirmation window asks you to continue.
8. Measure the paramagnetic reference and use its susceptibility to calculate the remenant magnetic field in the instrument.
9. Set the instrument field to oppose the remenant magnetic field.
10. Measure the paramagnetic reference again and calculate the net magnetic field.
11. Adjust the magnetic field again as necessary and repeat until the desired field is obtained, or until you cannot achieve better results.
12. Mount your sample to a sample holder and attach to a sample rod. *Be sure to measure the sample offset carefully.* Unless your sample is permanently magnetized, there will be no signal when you scan the sample to determine its location. You will have to use the "Enter Offset manually" option.

13. Warm the chamber to 300 K and insert your sample into the instrument, exposing it to 200 Oe of field in the motor. Specify its sample offset as closely as possible.
14. Set the instrument temperature or begin your measurement sequence. It is usually a good practice to measure the sample while you cool it in zero field and record the results to a data file to help determine the actual remanent field later.

4.5.3 Critical Temperature Measurements

The superconducting critical temperature of many elements are well-known. In fact, high purity indium samples are used in the factory to validate the instrument's thermometery calibration. The following procedure is used to perform this measurement and may serve as a model for similar measurement of other samples.

1. When measuring type I superconductors, minimize the remanent magnetic field first as described in the "Zero Field Cooling" section above. There will still be enough remenant magnetic field to generate a sizeable signal from a superconducting sample below its critical temperature. A minimal magnetic field is necessary to obtain an accurate measure of critical temperature.
2. Mount your sample to a sample holder and attach to a sample rod. *Be sure to measure the sample offset carefully.* Unless your sample is permanently magnetized, there will be no signal when you scan the sample to determine its location. You will have to use the "Enter Offset manually" option.
3. Create a sequence that contains a VSM Moment vs. Temp. command, starting above and ending below the expected critical temperature of the sample. Use a short averaging time, such as 0.5 sec., because the superconducting phase transition is usually very apparent. In this measurement, temperature precision is generally more important than moment measurement precison. Using a short averaging time allows you to measure continuously while changing the temperature and maintain a high data density with relatively small temperature uncertainty.
4. Run the sequence.
5. Once you know the critical temperature roughly, you may determine it more accurately by running another sequence over a smaller temperature range at a very slow sweep rate, such as 0.1 K/min.

4.6 Common Sources of Error

The following are the most common sources of measurement error:

4.6.1 Magnetic Field Errors

The reported magnetic field is derived from the net current that is known to be passing through the magnet solenoid. A correction is applied for the current that passes through the QuickSwitch while charging or discharging the magnet. However, the actual magnetic field at the sample location may be different than the reported magnetic field due to magnetic flux that is trapped inside the solenoid. This trapped flux occurs because electrical currents may indefinitely persist within the superconducting wire, which lacks electrical resistance, and generate a remanent magnetic field. This magnetic field error depends on the recent history of the magnet and is usually the most problematic when the reported magnetic field is very low, or zero. Use a known paramagnetic reference to determine the remanent field and post-process your data to correct for this error if necessary. Automating the magnet charging with a sequence to create a reproducible magnet history helps make the remanent field reproducible for a given measurement series. The remanent field will differ if you use different charging operations to set the same magnetic field. You may need to characterize the remanent magnetic field following all magnet charging operations in your sequence. Our experience clearly demonstrates the accuracy this procedure will achieve.

4.6.2 Sample Holder Effects

As described in the section above, the sample holder and sample securing agent may contribute to the measured signal. Measure each independently to determine the effects they have on measurements use centering scans to look for other print source dipole signals on the “blank” holder.

4.6.3 Sample Location Errors

Improperly-located samples will yield measurement errors. It is important to determine the correct sample offset before beginning measurements. If the sample temperature will be changed during or between measurements, it is important to adjust the sample location during the measurement process. Thermal contraction of the sample rod and sample holder will result in the sample location changing. Using the auto-tracking feature is the best way to automatically adjust the sample location. When utilizing the periodic centering scan option, instead, the sample location might not be adjusted for each reading, and noticeable discontinuities may appear in the data each time the sample location is adjusted.

4.6.4 Loosely Mounted Samples

When samples are not mounted securely to the sample holder they will not undergo smooth sinusoidal motion within the detection coils. The sample signal will be rejected as out-of-phase, or unknown measurement behavior will result, and the sample may potentially fall off of the sample holder and become lost. It is very important to mount samples securely for accurate measurements.

4.6.5 Temperature Errors

The sweep rate of the temperature will dictate the accuracy of the reported temperature as compared to the actual sample temperature. The analytical function should provide very reasonable accuracy for sweep rates up to 10 K/minute. As noted in the thermometry section of the manual, this instrument tries to always report the sample temperature, not just the thermometer reading. However, for very fast sweep rates, the user should validate the accuracy by comparison to a known response, like the Curie-Weiss behavior of dysprosium oxide.

4.6.6 Environmental Sources of Noise

The SQUID system can be sensitive to environmental sources of noise. The most concern is for broadband RF radiation, like that associated with arc welding or RF-induction furnaces. Electrical transformers can be a source of noise. There are indications that cell phones can also disrupt the SQUID signal.

Another concern is stray magnetic fields associated with charging of other magnets or the operation of items like an elevator. The full shield around the dewar will help mitigate these effects.

A simple technique to determine if the system is experiencing interference with external noise sources is to monitor the SQUID voltage as a function of time. Look for discontinuous changes in the SQUID voltage. While it is best to isolate and remove the source of noise, the VSM lock-in style measurement and other software features may be able to reject the interference. Please contact a Quantum Design service representative for more details on these procedures.

4.7 Further Reading

The following books and articles are a guide to general SQUID applications. Please see the references therein for more detailed issues.

- *Superconducting quantum interference device instruments and applications,* R. L. Fagaly, **Review of Scientific Instruments 77, 101101 (2006)**.

- *Demagnetizing Factors of the General Ellipsoid,* J. A. Osborn, **Phys**. Rev. 67, 351 (1945)

- *The SQUID Handbook: Fundamentals and Technology of SQUIDs and SQUID Systems, Volume I*
  John Clarke, Alex I. Braginski
  ISBN: 3-527-40229-2
  Hardcover
  409 pages
  August 2004

- *The SQUID Handbook: Applications of SQUIDS and SQUID Systems, Volume II*
  John Clarke (Editor), Alex I. Braginski (Editor)
  ISBN: 3-527-40408-2
  Hardcover
  653 pages
  October 2006

- SQUID Sensors: Fundamentals, Fabrication and Applications

  Series: NATO Science Series E: , Vol. 329

  Weinstock, H. (Ed.)

  1997, 720 p., Hardcover

  ISBN-10: 0-7923-4350-6

  ISBN-13: 978-0-7923-4350-9

A P P E N D I X A

Maintenance and Servicing

A.1 Overview

In this appendix we provide maintenance tips and instructions for user-serviceable items on your MPMS SQUID VSM. For help servicing the instrument, contact your Quantum Design service representative. A list of service representatives can be found on the Quantum Design web site at http://www.qdusa.com, or by calling the Quantum Design world headquarters in the United States at 1-800-289-6996.

Also, please refer to the supplied manufacturer's instructions for the vacuum pump, monitor, and printer.

A.2 Periodic Maintenance Checklist

To ensure proper instrument operation and long instrument lifetime, check the following items on the schedule shown.

Every six months:

- ❑ Inspect magnetic latches on sample rods and clean if necessary.
- ❑ Inspect bearing surfaces on sample rods and clean if necessary (do not use solvents.)
- ❑ Check pump oil level and add oil if necessary
- ❑ Check pump exhaust filter located at rear of pump console and drain if necessary.

Once a year:

- ❑ Clean the sample chamber
- ❑ Check serviceable o-rings for cracks and debris

Once every two years:

- ❑ Drain old pump oil and replace with fresh oil.

A.3 Service Procedures

You may perform the following service procedures on the instrument at the installed site. Do not perform other service procedures not described here unless expressly instructed to do so by a Quantum Design representative.

A.3.1 Recovering Lost Samples

If a sample falls off of the sample holder inside the instrument, recover it using a long rod, such as a rifle cleaning rod. Place double sided tape on the end and insert that into the sample chamber. **It is important that the tape does not come off the rod!** If you still cannot retrieve your sample please call Quantum Design for assistance.

If a powdered sample is lost inside the instrument, it is recommended that you clean the sample chamber using the procedure below.

A.3.2 Cleaning the Sample Chamber

The sample chamber may require occasional cleaning, especially if a lost sample has contaminated the chamber. If you know that magnetized material has contaminated the chamber, it is recommended that you remove the VSM head prior to cleaning the sample chamber, to prevent contaminating the VSM head (see separate procedure below.) Only isopropyl alcohol applied to a lint-free cloth may be used to clean the sample chamber. Use a rifle cleaning rod or similar device with a protective coating such as shrink tubing.

1. Set the chamber temperature to 300K and wait for stable 300K temperature to be achieved.
2. Set the magnetic field to zero and wait for zero field condition to be achieved.
3. Vent the sample chamber continuously (“Instrument > Chamber… > Vent Cont.”)
4. Wrap a lint-free cloth on the tip of a long rod such that the cloth is captured and may not be released inside the chamber. A rifle cleaning rod is ideal. Soak the lint-free cloth with isopropyl alcohol.
5. Insert the cloth into the sample chamber and slowly slide it all the way down. Do not force the cloth into the chamber if you feel much resistance—the cloth must not become lodged or stuck in the sample chamber!
6. Use a twisting motion to get the cloth to clean the sides of the chamber. Slowly remove the rod and replace the cloth with a clean one. Repeat until the cloth comes out clean.

A.3.3 Pump Oil, Changing and Filling

Only use the procedure outlined here if your MPMS SQUID VSM is not outfitted with the EverCool recondensing dewar option. Instruments with the EverCool option do not have oil-based pumps and require different pump servicing. See the EverCool user's manual for service procedures on EverCool equipped instruments. The procedure below ensures that the chamber and annulus are not in a vacuum when opening the pump to atmosphere. This prevents air from streaming in and contaminating these spaces while servicing the pump.

1. Set the instrument temperature to 300 K and wait for it to stabilize at that temperature.
2. Vent the sample chamber ("Instrument > Chamber… > Vent/Seal")
3. Shutdown the instrument ("Instrument > Shutdown… > OK")
4. Select "Utilities > Log Data…" and switch to the "Diagnostic Items" tab
5. If not already checked, check the box next to "Annulus Pres (Torr)" and monitor its value
6. Wait for the annulus pressure to reach 750-760 Torr.
7. Move items off of the pump cabinet, including the printer. Loosen the screws on the side of the console. The front cover and sheet-metal enclose are mounted together. To remove this assembly, lift the enclosure at the rear and carefully slide it towards the front before lifting it vertically. Place it into a safe spot to prevent damage.
8. Shut down the pump with the power switch on the pump.
9. Drain the oil from the exhaust filter into a cup or drain pan. The exhaust filter may be drained by turning the nipple on the bottom of the filter clockwise with your fingers. (Do not turn the entire clear filter cup unless it is necessary to replace the filter element. The cup has a removable seal that is best left untouched if possible.)

Note: The exhaust filter may be drained at any time as necessary, but should be checked whenever servicing the pump. It is not necessary to disconnect the pump to drain the exhaust filter.

10. If you are changing the oil, drain the oil from the pump into a cup or drain pan. The pump may be drained by turning the oil plug at the base of the pump counter-clockwise (just below the oil level view port.) If you only need to refill the oil because the oil level is low, skip to step 12.
11. Replace the drain plug and fill the pump with oil through the fill port on top of the pump. Fill only to the marking on the top of the oil level view port. Use only the oil supplied with the instrument. See Appendix C for re-ordering information.
12. Replace the plug in the fill port and turn the pump back on. Replace the cover on the pump cabinet.
13. Wait two minutes with the pump running before operating the instrument at all. This allows the pump to pump air out of itself and the pumping line.
14. Purge and seal the sample chamber ("Instrument > Chamber > Purge/Seal").

A.3.4 Cosmetic Panels, Removing and Installing

The cosmetic panels on the instrument may need to be removed in the following circumstances:

- To move the instrument through a doorway
- To access the magnet controller, gas handling controller, cryogen tanks (dewar), magnetic shielding, vibration isolation, cables, power distribution equipment, cryogenic insert, and other associated equipment for service

The panel in front of the magnet controller may be removed by opening the left cabinet door and pulling the panel straight forward. The panel snaps onto the cabinet in 3 locations. A similar panel on the right side of the cabinet attaches the same way, but rarely needs to be removed.

The other seven cosmetic panels are plastic and need to be removed and installed in sequence to gain access to the hardware in the lower half of the cabinet:

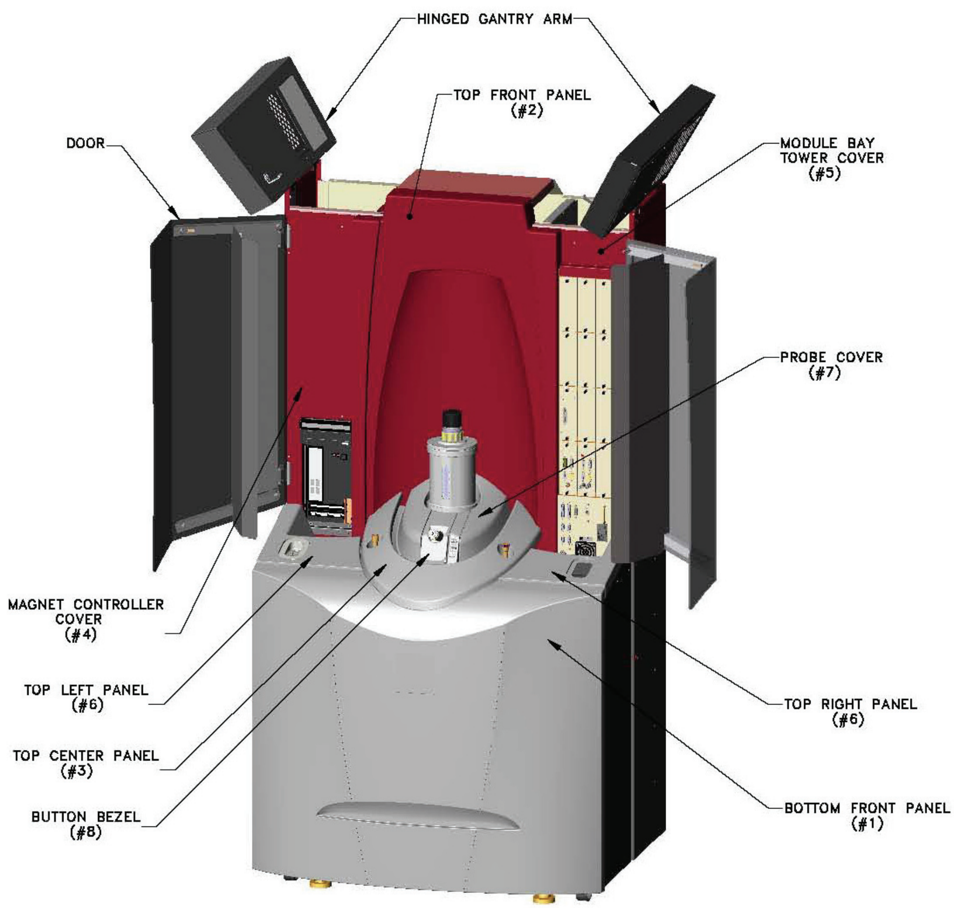


Figure A-1. Main cabinet cosmetic panels.

To remove all panels follow the sequence below:

1. **BOTTOM FRONT PANEL**

Pull bottom edge of panel away from cabinet (there are snaps on either side), swing bottom edge outward, then lift entire panel up, out of lip in top panels.

To move the cabinet through a doorway, this is the only panel that needs to be removed. To access the equipment above the dewar, continue removing the top panels:

2. TOP FRONT PANEL

Open both doors and swing open (upward) the hinged arms above each door to remove the locking pins that secure this panel. Then lift the panel upward, over the VSM head (motor) and away from the cabinet. (Notice how the bottom of this panel sits in notches on the top left and right panels (#6)).

3. TOP CENTER PANEL

Lift directly upward (tabs on either side locate this panel in the top left and right panels (#6)).

4. MAGNET CONTROLLER COVER

Open left door and carefully pull the panel towards you (it snaps to the cabinet in three locations).

This provides access to the magnet controller and the computer.

5. MODULE BAY TOWER COVER

Open right door and carefully pull the panel towards you (it snaps to the cabinet in three locations).

6. TOP LEFT AND RIGHT PANELS

Remove covers #5 and #6 first, open doors, move cabling out of the way, and lift panels straight up (they snap to the sheet metal cabinet in three locations).

This provides access to most of the serviceable equipment. Should you require further access, the remaining panels are attached to the cryogenic insert (probe):

7. PROBE COVER

Carefully spread the two front-facing edges of the cover and lift straight upward.

8. BUTTON BEZEL

This panel rarely needs to be removed. It has a sheet metal frame that is secured to the top of the cryogenic insert with four screws. You may remove the screws and carefully slide the panel forward over the helium fill tube. You will need to carefully disconnect the wiring to the buttons and the magnet current leads.

When reinstalling the panels, reverse the order of operations.

A.3.5 VSM Head, Removing and Reinstalling

It is sometimes necessary or desirable to remove the VSM head (motor) from the system. For example, to clean magnetized contamination out of the sample chamber, you may need to remove the VSM head to avoid contaminating it with the magnetized debris. Follow the steps below to remove and re-install the VSM head.

1. Remove any sample and sample rod from the chamber with “Sample > Install/Remove…” Select the “Open Chamber” button and wait for the system if necessary. Then remove the sample rod.

2. Select the "Shutdown" button. This disables the VSM head.

3. Remove the following plastic panels: top front panel, top center panel, probe cover. See steps 2, 3, and 5 in the section "Cosmetic Panels, Removing and Installing." You may skip steps 1, 4, and 6 in that section.

4. Disconnect the round cable connector on the back of the VSM head by pulling straight out.

5. Make sure to have the VSM head stand open and close by. This stand is part of the supplied instrument packaging, and is covered by a wooden box with metal clasps and metal handles on two sides.

6. Turn the locking nut under the VSM head counter clockwise while holding the VSM head. The head is heavy and you will need to stabilize it.

7. Lift the VSM head straight up and place in the stand. **Never lay the VSM head on its side**—this can damage delicate suspension springs in the head.

To replace the VSM head.:

1. Position the VSM head over the sample manifold block on top of the cryostat and carefully set it in the center. Rotate until the armature position window faces the front of the instrument.

2. While holding the VSM head, turn the lock nut clockwise until tight.

3. Reconnect the cable by plugging it straight in.

4. Select the "Back" button on the Install/Remove Sample Wizard.

5. Select the "Open Chamber" button again. This restarts the VSM head.

6. Replace the plastic panels in the reverse order you removed them.

A.3.6 Moving the Instrument

The main instrument cabinet has four casters (pivoting wheels) and may be easily rolled from one location to another. However, the weight of the cabinet normally does not rest on the front casters, but instead on the rear casters and on leveling feet mounted just in front of the front casters. This prevents accidental instrument movement.

To move the instrument first remove bottom front panel, as described in the section above. Then lift the front corners of the cabinet just enough to remove the weight from the leveling feet and screw the leveling feet up, off of the ground with your fingers. Use a piece of scrap wood or metal pipe as a lever to perform this lifting operation. You will need to lift near the front corners, where the cabinet frame is located, rather than near the center, where the vibration isolation and cryogen tank (dewar) is located. Once the leveling feet are screwed up and out of the way, allow the cabinet to rest on all four casters and roll it to its new destination. With the bottom front panel removed it should fit through a standard doorway.

Take care not to stretch or stress the hose and cables running between the main cabinet and the pump cabinet when moving the instrument. You should have someone else help you by rolling the pump cabinet along side the main cabinet.

Make sure that the instrument is properly leveled again at its new destination.

A.3.7 Air Filters, Cleaning or Changing

The air filter on the front, at the bottom of the module tower filters the air cooling the module bay power supply. It may be removed by removing the four screws holding the frame in place in front of the fan. Clean the filter with mild soap and water to remove dust and debris, and air dry.

Do not run the system for more than two days without the air filters in place.

A.3.8 O-rings, Cleaning or Changing

The various O-rings in the system may need to cleaned and re-greased occasionally. Use only isopropyl alcohol applied to a lint-free cloth to clean O-rings. After removing any O-ring, inspect it for cracks or other damage and clean the O-ring groove and mating surfaces with a lint-free cloth and isopropyl alcohol. Use a small amount of silicone vacuum grease to lubricate and lightly coat the O-ring completely. The O-ring should just look wet, but no grease itself should be visible or discerned as a lump or layer. Reinstall the O-ring only if it is not cracked or damaged. The utility kit supplied with your system contains spares of the most commonly replaced O-rings. Contact your Quantum Design service representative to replace cracked or damaged O-rings.

A.3.9 Replacing Fuses

The system has several user accessible fuses. In case you encounter situations where a fuse blows or a breaker jumps, contact your Quantum Design service representative to allow investigating the reason and provide advice.

Laboratory power outlet:

According to Quantum Design instructions and for safety reasons, the laboratory power outlet has to be equipped with 16A breaker.

Power Distribution Unit:

The main power entry located on the power distribution unit at the rear of the system has a 10A resettable breaker.

The Module Bay Tower (model EM-QA) utilizes two 6A, 20 mm delay fuses at the power entry. These should only be replaced with equivalent fuses and should always **both** be replaced at the same time. Spare fuses can be found in your utility kit.

The Magnet Controller (model EM-QB) utilizes two 8A, 20 mm delay fuses at the power entry. These should only be replaced with equivalent fuses and should always **both** be replaced at the same time. Spare fuses can be found in your utility kit.

To access the magnet controller you will need top remove the magnet controller cover, as indicates in section A.3.4.

A.3.10 Unplugging Nitrogen Exhaust

There are cases when the nitrogen exhaust mechanism of the SQUID VSM fails due to plugging, presumably by air leaking into the dewar. If the rear, external exhaust fails to operate properly, this situation must be remedied before a transfer of more liquid nitrogen. While the front fill and exhaust ports for the nitrogen are a safety release, they should not show venting during normal transfers. If there are limited exhaust capabilities, an overpressure in the nitrogen tank can lead to a catastrophic failure of the dewar and damage to the probe.

The primary advice is to verify there are no leaks into the nitrogen dewar. Check that both front exhausts have the proper o-rings in place and are secure. Kink the rubber tubing coming from the rear exhaust and allow a slight pressure to build up such that releasing the kink results in a burst of nitrogen gas. This test would verify the rear pressure relief valve is operational and the exhaust is at least somewhat clear. If during the start of the transfer the rear exhaust does not open within the first 30 seconds, then stop transferring.

It is recommended to transfer the nitrogen and helium on separate days. Since there is no nitrogen level meter and the presence of nitrogen is critical for low temperature operation and minimizing consumption of helium, the nitrogen should be added once a week. Do not use a high pressure (~200 psi, ~14 bar) liquid nitrogen storage dewar, but instead stay with the lower pressure (22 psi, 1.5 bar) rated nitrogen containers. The helium should be added as needed.

It is well known that for nitrogen jacketed dewars, the transfer of helium will cause cooling of the nitrogen tank such that a vacuum is pulled. Even though there is plenty of nitrogen in the tank, this vacuum can lead to accumulation of ice inside the exhaust ports if there is a leak or if the nitrogen ports are opened shortly after a helium transfer. Normally, this situation will return to equilibrium within a few hours after the transfer of helium and the nitrogen gas boil-off will exhaust as expected. However, there could develop situations where the exhaust ports are frozen open or leak in such a way as to generate a large enough ice plug that completely prevents the exhaust port from working. The solution is to clear the ice plug, find the source of leak and fix it.

Do not confuse the accumulation of ice on the outside of the exhaust lines with a plugging of the exhaust. With high boil-off rates, ice is expected to appear, especially near the top of the dewar, under the foam insulation.

Contact customer service for additional guidance and the procedure for establishing proper flow out the rear exhaust.

A.4 Initial Cooldown Procedure

The initial cooldown procedure is required after the system ran out of cryogens and warmed up for an extended period of time. Typically at that stage the probe (including magnet) and dewar are at room temperature.

Before starting the cooldown procedure you will need 100 liters of liquid helium, 100 liters of liquid nitrogen and a helium gas bottle with regulator. Allow about 3-4 hours for cooling the system down from room temperature.

If you have any questions contact your Quantum Design representative for assistance.

A.4.1 Preparing the System

1. All cables are connected to the probe with one exception: Disconnect the SQUID control cable (3501-500) from the SQUID module (model CM-F) plug JF-1.
2. The pump hose is connected to the pump.
3. Start *MultiVu* (the initial starting of *MultiVu* will be longer due to the software trying to tune the SQUID which is temporarily disconnected).
4. Start a log file (e.g., Date_Cooldown.dat). Select all items in each tab.
5. Start *GHMagnaMon*: Open the tools folder from the Desktop and start the *GHMagnaMon* utility software. To initialize it click the **Init Can** button followed by the **Readings** button. All valve icons should be red (closed).
6. Start the pump and let it warm up for 5 minutes.

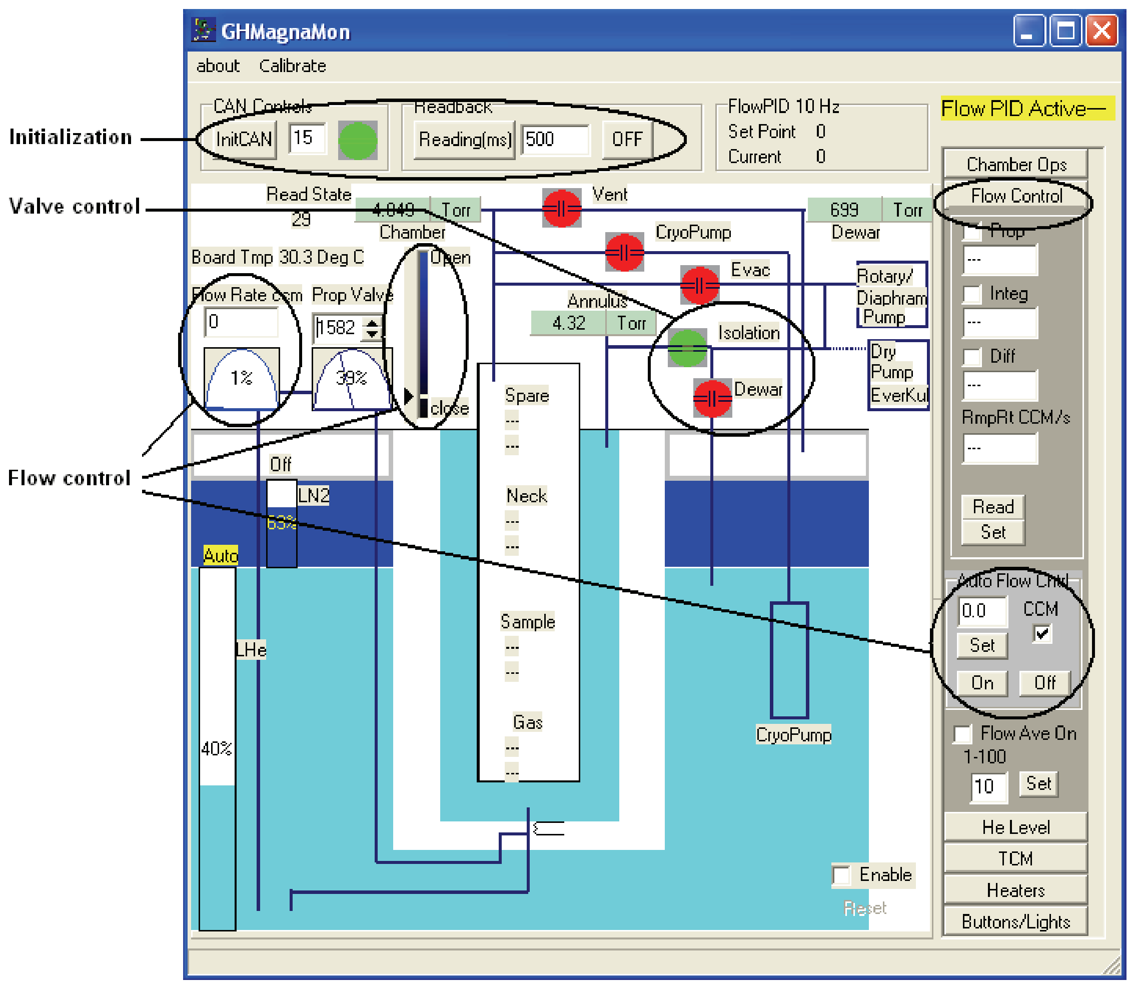


Figure A-2. GHMagnamon utility.

A.4.2 Purging the Dewar with Clean Helium

1. Install the modified fill port adapter with the hose barb on the helium fill port (the adapter is provided with the utility kit).
2. Connect a He gas bottle to the adapter.
3. Close valve at helium bottle regulator and click on the **Dewar** valve in *GHMagnaMon*. Valve icon should turn green (open).
4. The Dewar is now being pumped-out. Pump for 2-3 minutes.
5. Click again on the **Dewar** valve. Valve icon should turn red (close).
6. Open helium bottle valve (0.5 bar max) back fill the dewar until gas exits the relief valve at the top plate.
7. Repeat purging 2 times.

A.4.3 Flushing the Probe with Clean Helium:

1. Select **Flow Control** in side bar menu.
2. Select the "**CCM**" tag in the side bar.
3. Click the **Isolation** valve to start pumping on the annulus. Valve icon should turn green (open).
4. Open and close the proportional valve by moving the **slide bar** all the way up and down.
5. Repeat 3 times and leave valve fully open.
6. The flow should increase to over 300 cc/min.
7. Continue flushing for 15 minutes.
8. Enter a value of 50 for the **flow rate** and select **Set.**
9. Select **ON** to start flow control.
10. Keep *GHMagnaMon* running in the background.

A.4.4 Filling Liquid Nitrogen and Liquid Helium:

1. Connect nitrogen hose to LN2 inlet and start filling.
2. Get helium storage dewar ready with transfer line.
3. Make sure helium gas bottle is closed. Quickly unscrew adapter from helium inlet port and insert transferline. Start filling the dewar with helium.
4. Connect the helium bottle to the storage dewar. Pressurize the storage dewar to provide approx. 3 lpm flow.
5. In *MultiVu* start the helium fill utility to monitor the helium level.

6. Fill LN2 until tank is full (liquid nitrogen will spit out of the rubber exhaust hose. CAUTION: liquid nitrogen could crack your lab floor. Ensure proper protection).
7. Fill LHe to about 60-80%. If the storage dewar runs dry stop immediately.

A.4.5 Verifying Probe is Working:

1. Exit GHMagnaMon.
2. Reconnect the SQUID control cable (3501-500) to the SQUID module (model CM-F) plug JF-1.
3. In MultiVu tune the SQUID: In Utilities/SQUID open **squid control** and select **Auto Tune**.
4. In MultiVu set the temperature to **298K**.
5. Install the Pd sample and set the field to **1 Tesla**.
6. Locate the sample and verify moment calibration (see section 3.9.2).
7. Run a sequence down to base temperature. Note: Running for 1.8K right after an initial cooldown might take more time than usual. Allow some time for the system to settle.
8. Install all cosmetic panels.

A P P E N D I X B

Advanced Software Operations

B.1 Overview

In this appendix we describe advanced operations performed with MultiVu. Section B.2 outlines the SQUID range settings. The following sections describe specific diagnostic tools. This is crucial in case troubleshooting is required. Section B.3 focuses on diagnostic data logging, section B.4 on the background event log and section B.5 on specific SQUID diagnostics. Finally in section B.6 we describe how to reset the magnet.

B.2 Squid Ranges and Autoranging

The SQUID ranges are labeled 1, 10, 100, and 1000. This corresponds roughly to the multiplier required to convert the output voltage from the SQUID electronics to the number of flux quanta (Φ_0) measured by the SQUID. As the output voltage from the SQUID electronics is in the range ±5V this means that the maximum SQUID signal one can measure in each of the ranges is roughly $\pm 5\Phi_0$, $\pm 50\Phi_0$, $\pm 500\Phi_0$, and $\pm 5{,}000\Phi_0$. However, the maximum full-scale reading in emu (or A-m2) depends on numerous calibration factors. You may simply remember that 1 is the most sensitive range, and that each successive range reduces the amplification by x10.

The instrument is calibrated to minimize discontinuities between ranges; however, discontinuities cannot be eliminated completely. It may be possible to notice the instrument range changes as small steps or discontinuities in your data. The SQUID range is one data item that is collected with each data record, so it is possible to examine your data and know whether a range change correlates with a discontinuity in magnetic moment data.

The SQUID range selection appears on the “Advanced” tab of the measurement and sequence command windows. The default setting is sticky autorange.

B.2.1 Sticky Autorange

Change the SQUID range only if the signal is < 9% or > 90% of the current full scale range. This setting minimizes the amount of time spent changing ranges and minimizes the number of possible data discontinuities due to range changes.

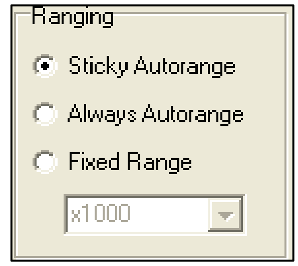


Figure B-1. SQUID ranging.

B.2.2 Always Autorange

Check the signal level during every single measurement and adjust the range for maximum signal every time. Signal may not exceed 90% of full scale. This setting maximizes the signal level, but also creates potential delay determining the best range setting for every data point and allows for data irregularities when the signal level is very close to the range boundaries.

B.2.3 Fixed Range

Keep the SQUID set to the selected range. Note that the system will not collect any data points with this setting if the signal exceeds the maximum of the selected range.

B.3 Logging Diagnostic Data

The menu command "Utilities > Log Data…" is used to log diagnostic data for the instrument. To use the logger, click "Browse" and specify a file name to write to. You may specify a new file name or an existing file name. Be careful: If you check the "Overwrite Existing File" box, any existing data in the file will be deleted! Also, specify how often to log data records to the file. Some problems are best diagnosed by very rapid data logging for a short period of time, while others are best diagnosed with very long logs with infrequent data records. A Quantum Design representative can usually help you determine the most appropriate logging interval to use when troubleshooting.

When you click "View Data," the specified data file will be opened in a graph view. But no data will be present in the file until you select data items to log from the other three tabs—"Standard Items," "Diagnostic Items," and "Advanced Items"—and until you click "Start" (begins the logging) or "Acquire Once" (records all of the checked data items one time only.) Once logging, data will continue to be recorded to the file at the specified rate until you click "Stop." You may change the data items being logged while the logger is running. A time stamp always accompanies each data record.

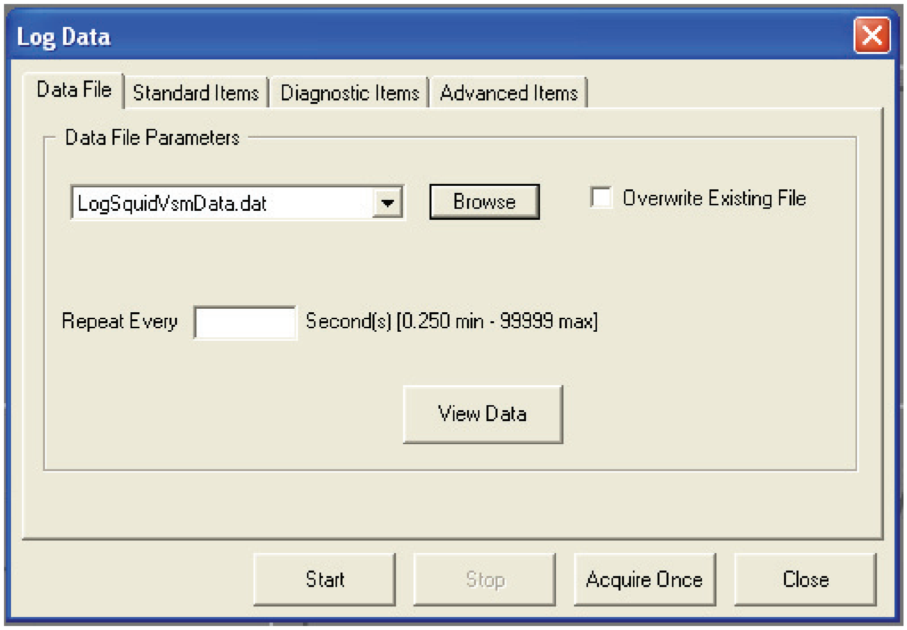


Figure B-2. Data logging interface.

B.3.1 Standard Items

Standard Items include basic information generally available in the MultiVu status bar. The state of the sample chamber, temperature control, field control, and motor are recorded in the data file as integers. The log data window displays the meaning of each of these status codes in real time, but this text is not included in the log data files. (Use "Utilities > Status Calculator…" to decipher the status codes. The "Query" button will decipher the instrument's current state.)

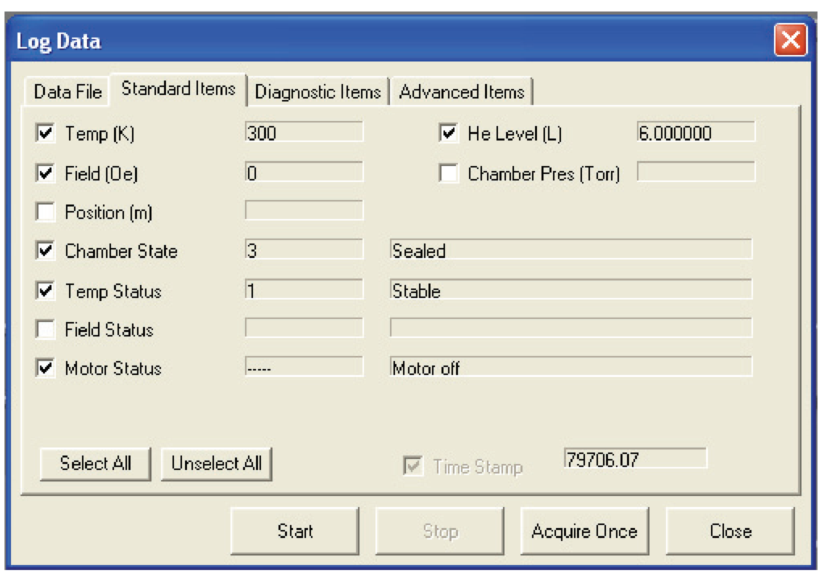


Figure B-3. Selection of data to log.

B.3.2 Diagnostic Items

Diagnostic Items include more detailed information about the data from each individual sensor or transducer in the system.

B.3.3 Advanced Items

Advanced Items are all configurable data items. A large amount of raw instrument data is available for logging when required. Configuration of these data items is accomplished with a map file which is usually supplied by a Quantum Design representative.

The menu command “Utilities > Sigma Log Data…” contains very similar capabilities as the log data command, except that you may log the statistics (stats) average and standard deviation of each data item as well. This generates data files that are significantly larger and more tedious to navigate than the standard log data command. The average values recorded with this command may be a simple average of all new data, or a running average of the last several data items, in which case each average may be calculated with some of the same data as the previous data record.

The log data and sigma log data commands have counterpart sequence measurement commands which may be executed within a sequence to start and stop data logging, change the log file or logging rate, or change the data being logged.

B.4 Using the Event Log

The event log is a text file containing diagnostic messages generated by the instrument, with time stamps. The event log is limited to 10 MB in size, and will begin over-writing itself when this limit is reached. It is arranged chronologically.

You may specify the different level of diagnostic messages that are displayed in the MultiVu event log viewer. Message levels range from informational to fatal errors. Access the event log with the menu command “Utilities > Event Log.” If you experience a problem with your MPMS SQUID VSM, you may be instructed to read the contents of the log to help determine the source of the problem.

Anytime an event occurs (with a level which is enabled in the Event Log viewer), a little dialog box with the three most recent events will be displayed. This ensures that you don’t miss potentially significant error messages.

B.5 SQUID Diagnostics

SQUID VSM MultiVu contains a diagnostic SQUID interface which allows you to change the SQUID bias, range, and offset. In addition, it allows you to quench the SQUID and the SQUID input circuit, eliminating standing currents by heating them above their critical temperature. You may also zero the output, tune the SQUID, and determine the transfer fraction (V/Φ_0). The "Output" selection allows you to control what signal is present at the BNC connector (JF-5 and JF-7) on the front of the SQUID control module, model CM-F.

These procedures are rarely necessary under normal instrument operation. They exist for use by service personnel and engineers. The SQUID diagnostics are accessed with the menu command "Utilities > SQUID."

B.6 Resetting the Magnet

Because it poses no resistance to the motion of electrons, the superconducting solenoid in the MPMS SQUID VSM tends to "trap" magnet flux. It can be very difficult to drive the magnet to truly zero magnetic field. Even though there is no net electrical current through the solenoid and no voltage across it according to the magnet controller, small currents may still circulate within the superconducting material.

Resetting the magnet is one way to help minimize the magnetic field or field gradiants in the sample chamber. The operation is completely automated, but should only be initiated by a user who understands the operation. The software first drives the magnet to zero field and then applies heat to the magnet with an integrated heater to drive the superconducting solenoid windings above their critical temperature. After resetting the magnet, some time is required for the magnet to cool back down to operating temperature. So you should wait a couple of minutes before charging the magnet again.

Reset the magnet with the menu command "Utilities > Reset Magnet." A confirmation window will follow.

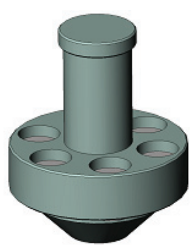
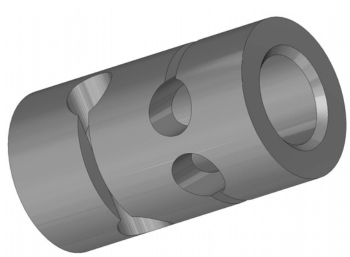
Note that resetting the magnet can use a significant amount of helium boil-off. For most purposes simply oscillating the field to zero from a field of a few tesla will provide a similar end result while requiring significantly less liquid helium.

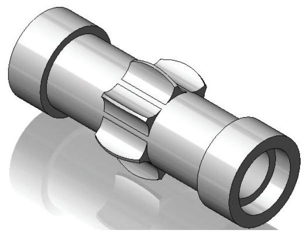
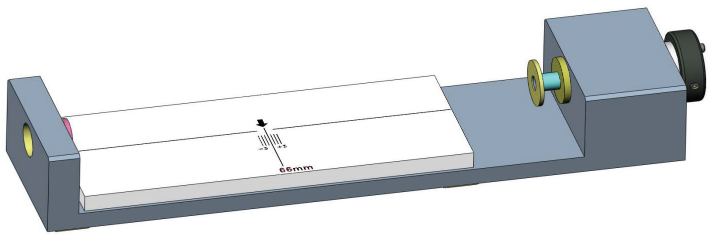
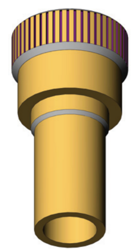
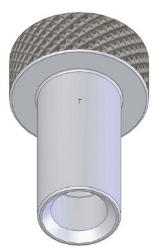
APPENDIX C

Ordering Replacement Parts

C.1 Overview

In this appendix we list the Quantum Design part number for loose or consumable parts you may wish to order throughout the lifetime of the MPMS SQUID VSM. Contact your local Quantum Design representative to order parts.

Picture	Name	Part Number
	quartz sample holder	4500-604
	brass sample holder	4500-608
	palladium reference (mounted)	4500-612
	sample rod	4500-600
	magnetic latch for sample rod	4096-358
	sample rod flexure	4500-607

	Sample rod bearing	4500-603
	sample mounting station	4500-620
	ultratorr fitting (tube fitting) for helium transfers	YSS-6-UT-A-8BT
	nitrogen fill port plug	4504-032
	pump oil	HH-110-25-012
	activated alumina for oil mist filter on rotary vane pump	H026-00-050
CONSULT A QUALIFIED QUANTUM DESIGN SERVICE REPRESENTATIVE BEFORE REPLACING ANY O-RINGS OR SEALS.		
	teflon gas cap seal used on helium inlet plug	4504-002
	o-ring for nitrogen fill ports	VON2-012

	o-ring for transfer line bayonet	VON2-014
	o-ring for top of sample space manifold	VON2-020
	o-ring for motor cap	VON2-022
CONSULT A QUALIFIED QUANTUM DESIGN SERVICE REPRESENTATIVE BEFORE REPLACING ANY FUSES		
	fuses for module tower power entry: 6.3A, 20mm, delay (external)	FD6.3-20MM
	fuses for magnet controller power entry: 8A, 20mm, delay (external)	FD8-20MM
	fuses for module bay QD-CAN outlets: 5A, 20mm, delay (external)	F5-20MMSB
	fuses for 5V power output in computer: 3.15A, 20mm, delay (internal)	FD3.15-20MM

A P P E N D I X D

System Specifications and Interconnects

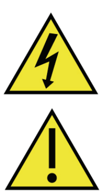
D.1 Overview

In this appendix we provide information on system specifications, such as power requirements and operating conditions (section D.2). We further provide interconnect diagrams for a more detailed understanding of the system architecture (section D.3).

D.2 System Requirements and Operating Conditions

Quantum Design service representatives will perform the initial installation of the system. These are however various requirements which need to be fulfilled prior to the installation.

D.2.1 Power Ratings



Voltage:	200-230 VAC, 50/60 Hz, single phase
Current:	typical operation 6-8 A
Mains Fusing:	10 A breaker at main power switch

D.2.2 Laboratory Power Outlet Setup

Laboratory power outlet is required to be fused to 16A MAX.

The Power cord supplied with the system is 2.5m (10 ft) long. The power outlet should be within that distance from the planned system location or an extension cord will be required (not supplied by Quantum Design).

Laboratory outlet specifications:

International industrial type connector
IEC 60309 6H Female
16A/250V
2P+E
I44 (splash proof)
Color Code: blue
Safety approvals: UL/CSA, VDE, IEC

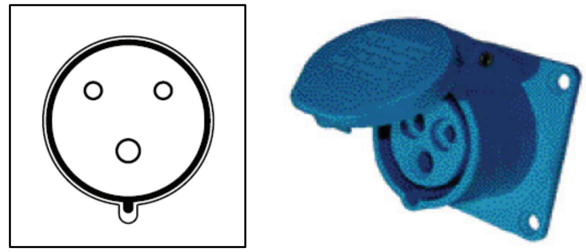


Figure D-1. Laboratory Power Outlet.

D.2.3 Laboratory Ambient Conditions

Operating ambient temperature: 10-40 °C (50-104 °F)
Operating ambient relative humidity: 10-90% (non condensing)

D.2.4 Cryogenic Requirements

For the initial cooldown the system requires the following quantities of cryogens:

Liquid Nitrogen: 100 liters
Liquid Helium: 100 liters (another 100 liters should be available as a back-up)
Pressurized Helium gas cylinder with regulator

Under normal operating conditions the cryogen holdtimes are typically 12 days for helium (100% = 70 liters) and nitrogen (100% = 55 liters). See section 1.5 for more details on transferring cryogens.

In case the system warms up, follow the cooldown procedure described in Appendix A.4.

D.3 System Interconnects

The various system interconnects are shown below to help understanding the general architecture of the system. Most of these interconnects do not need to be serviced by the user at all.

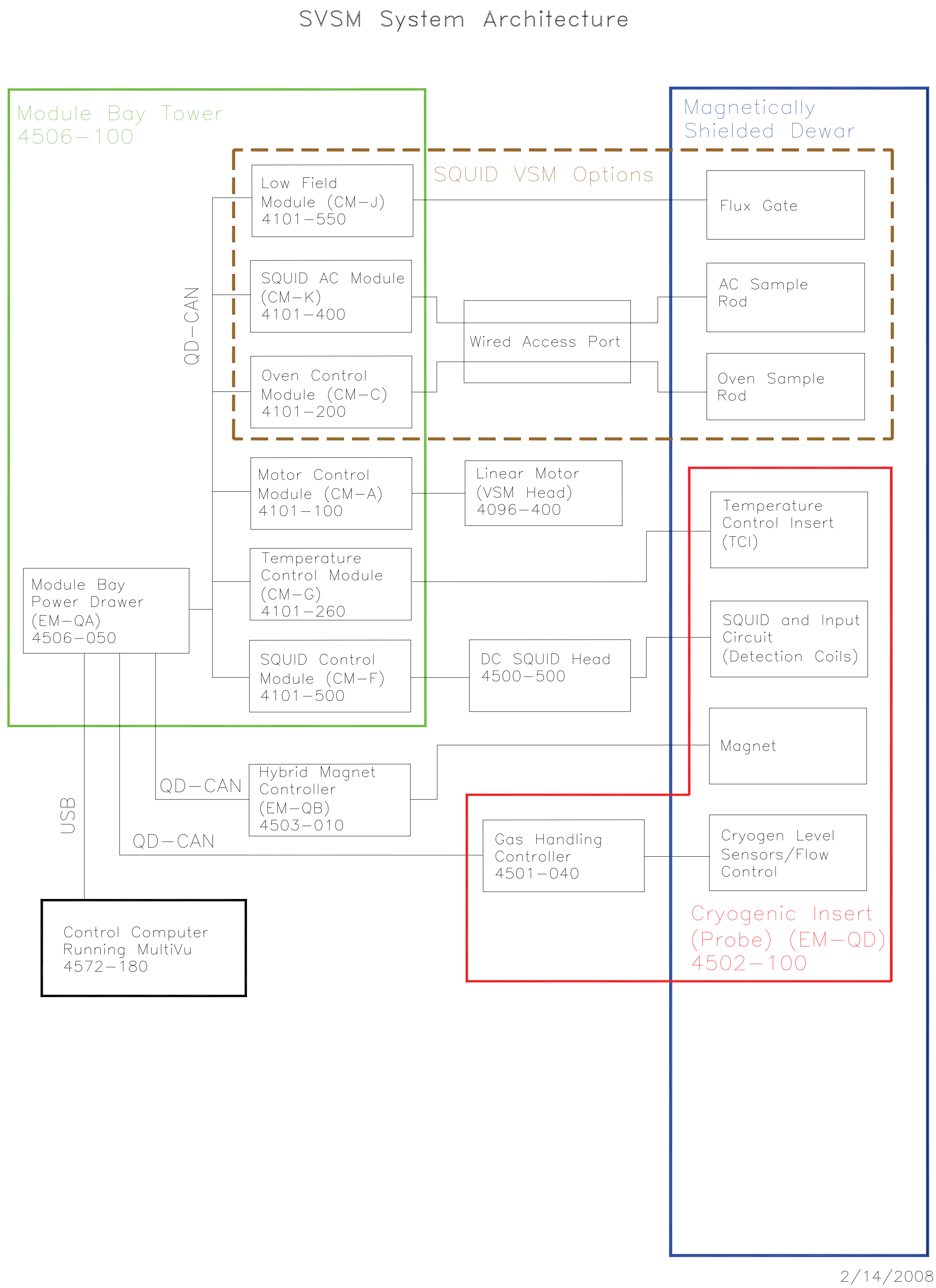


Figure D-2. SVSM System Architecture

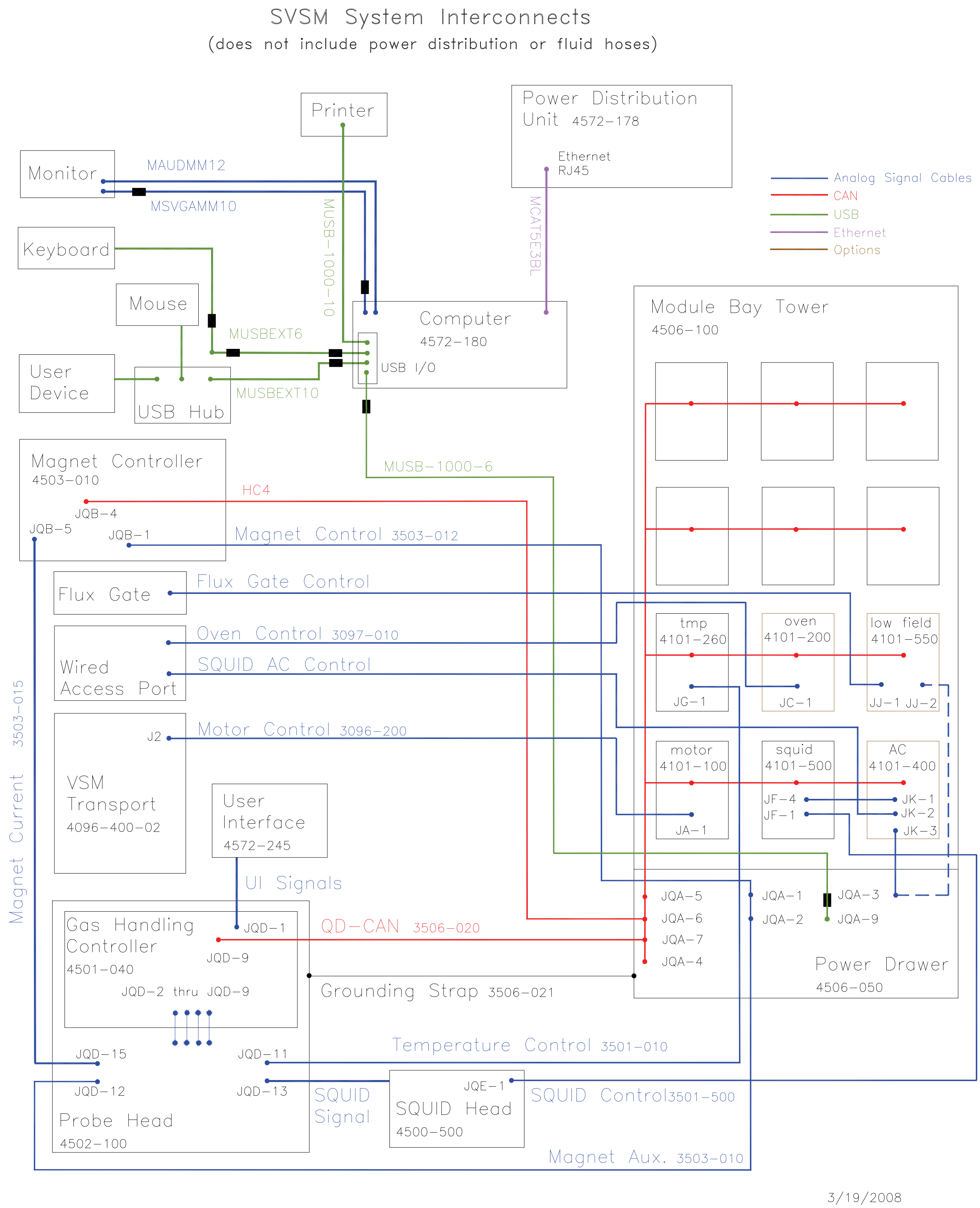


Figure D-3. SVSM System Interconnects

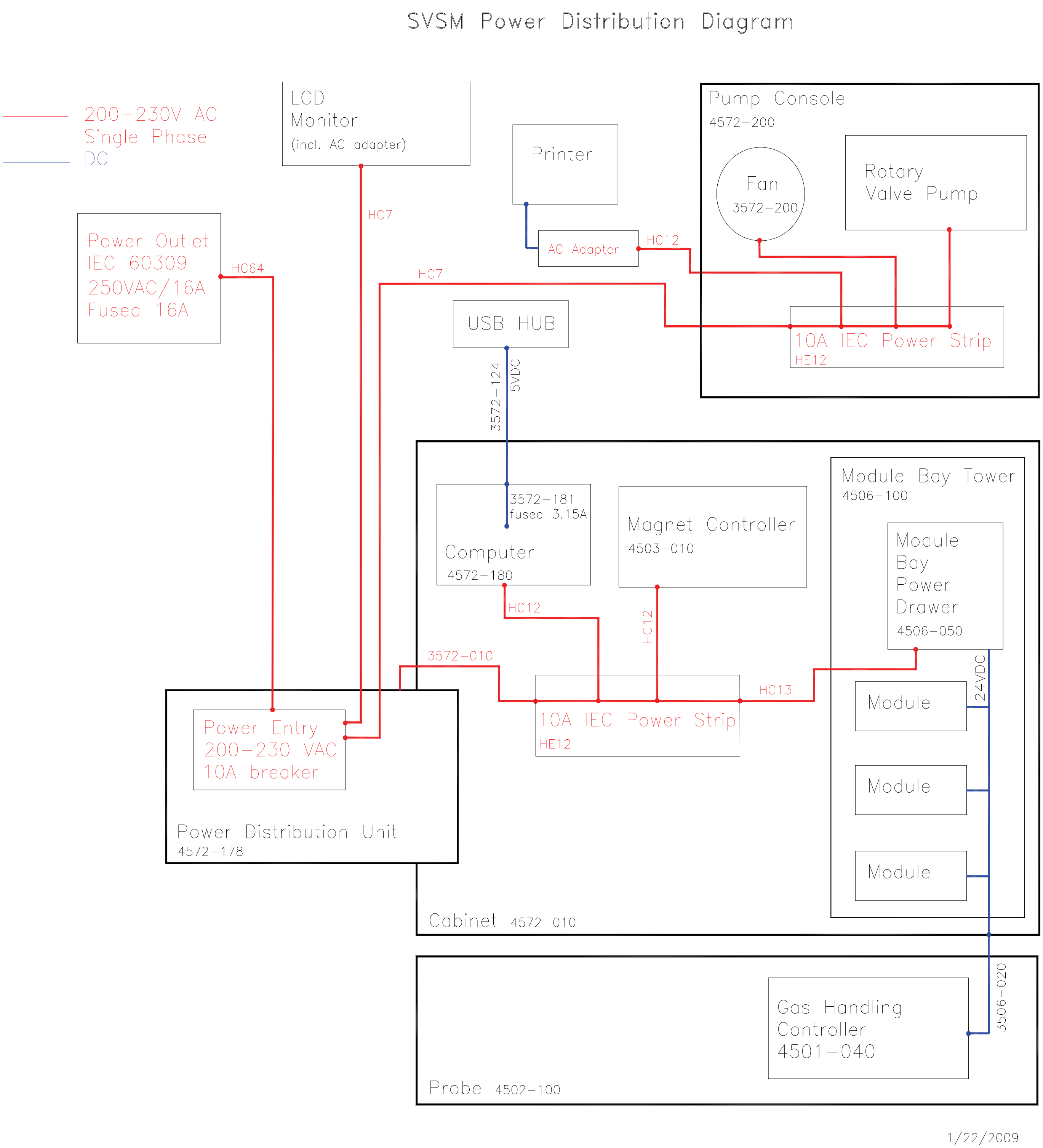


Figure D-4. SVSM Power Distribution Diagram

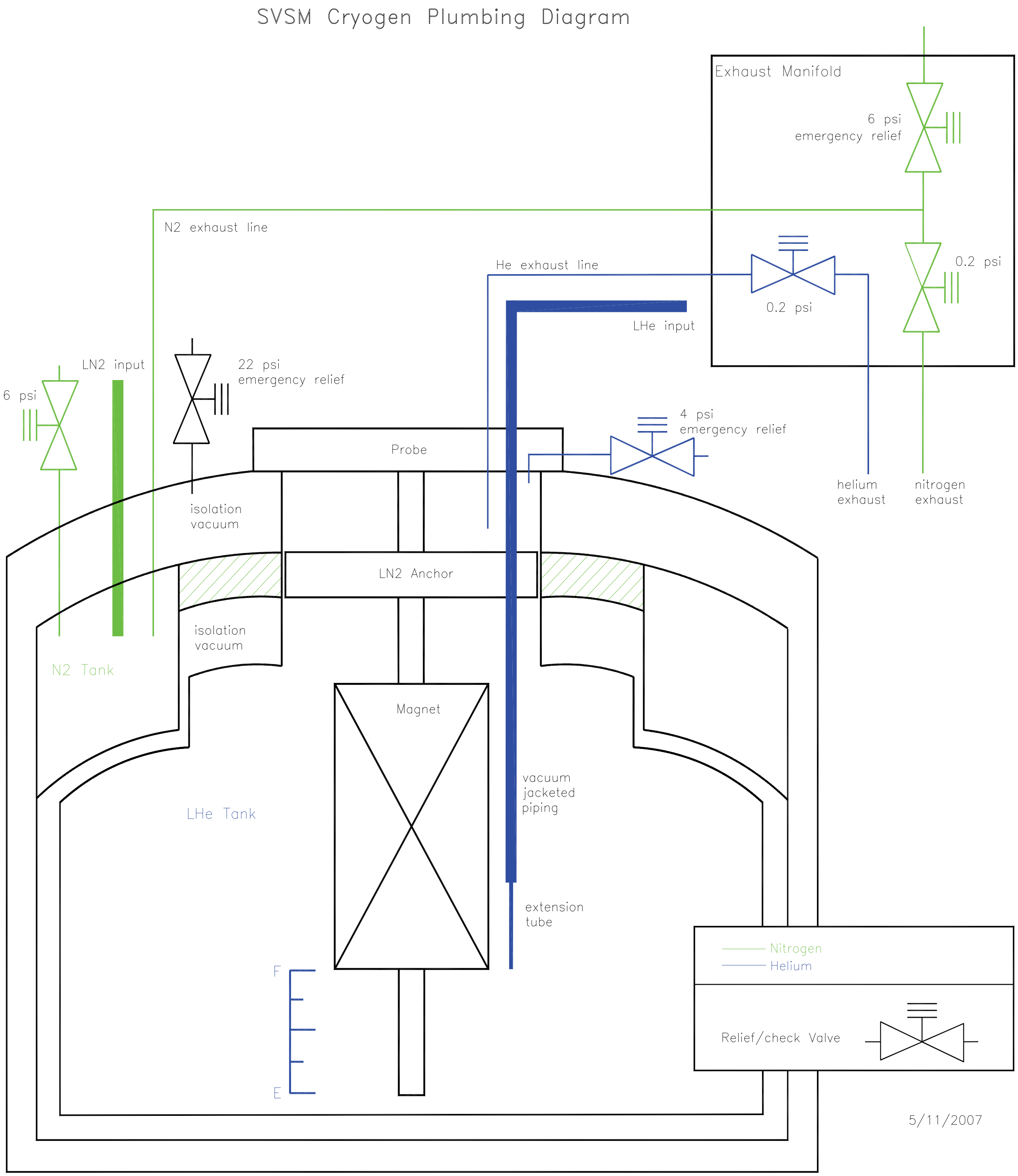


Figure D-5. SVSM Cryogen Plumbing Diagram