

**Q u a n t u m D e s i g n**

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# **Magnetic Property Measurement System<sup>®</sup>**

## **MPMS 3 User's Manual**

**Part Number 1500-100, F1**

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## **U.S. Patents**

5,053,834 High Symmetry DC Squid System  
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  
8,384,504 Superconducting Quick Switch.

## **Foreign Patents**

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

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# Contents and Conventions

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## P.1 Overview

**In this chapter** we describe the scope of the manual, the conventions used and most importantly the safety guidelines. The MPMS 3 uses cryogenics and high power components, we strongly recommend being aware of all hazards in order to prevent injuries and system damage.

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 illustrates and describes conventions that appear in the manual.
- Section P.4 describes the safety guidelines and regulatory information in the manual.
- Section P.5 gives disposal information and RoHS and WEEE compliance.

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## P.2 Scope of the Manual

This manual discusses the MPMS 3. 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 MPMS 3 MultiVu software.

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## P.3 Conventions in the Manual

<b>File menu</b>	Bold text identifies the names of menus, dialogs, options, buttons, and panels used in the MPMS 3 MultiVu software.
<b>File &gt; Open</b>	The > symbol indicates that you select multiple, nested software options.
<code>.dat</code>	The Courier font indicates file and directory names and computer code.
<b>Important</b>	Text is set off in this manner to signal essential information that is directly related to the completion of a task.
<b>Note</b>	Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances.



### WARNING!

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



### WARNING!

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



### WARNING!

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).



### WARNING!

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).



### WARNING!

This symbol signals strong magnetic fields. Used to indicate potential danger from a charged magnet.



This symbol signals information on **fusing**.



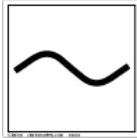
### PROTECTIVE CONDUCTOR TERMINAL

The protective conductor terminal symbol in the left figure identifies the location of the bonding terminal, which is bonded to conductive accessible parts of the enclosure for safety purposes.



### EUROPEAN UNION CE MARK

The presence of the CE Mark on the equipment signifies that it has been designed, tested and certified as complying with all applicable European Union (CE) regulations and recommendations.



### ALTERNATING VOLTAGE SYMBOL

This international symbol indicates an alternating voltage or current.



### STANDBY SYMBOL

The power standby symbol indicates a sleep mode or low power state. The switch does not fully disconnect the device from its power supply, depressing the button switches between on and standby.



### WASTE ELECTRICAL AND ELECTRONIC EQUIPMENT (WEEE)!

This symbol on the product or on its packaging indicates that this product must not be disposed of with regular waste. Instead, it is the user's responsibility to dispose of waste equipment according to local laws. The separate collection and recycling of the waste equipment at the time of disposal will help to conserve natural resources and ensure that it is recycled in a manner that protects human health and the environment. For information about where the user can drop off the waste equipment for recycling, please contact your local representative. Contact Quantum Design for instructions on how to disassemble the equipment for recycling purposes.


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## 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 MPMS 3 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.

	<b>WARNING!</b>
<p>If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired. Do not position the equipment so that it is difficult to operate the disconnecting device.</p>	

Observe the following safety guidelines when you use your system:

### P.4.1 Inspection for Damage

The MPMS 3 is carefully packaged at the factory to minimize the possibility of damage during shipping. Inspect the box for external signs of damage or mishandling. Inspect the contents for damage. If there is visible damage to the instrument upon receipt, inform the shipping company and Quantum Design immediately.

	<b>WARNING!</b>
<p>Do not attempt to operate this equipment if there is evidence of shipping damage or you suspect the unit is damaged. Damaged equipment may present additional hazards. Contact Quantum Design technical support for advice before attempting to power on and operate damaged equipment.</p>	

### P.4.2 Electricity

- 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. Permit only qualified electricians or Quantum Design personnel to open electrical enclosures and perform electrical servicing and checks.
- 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 C.2.
- To prevent damage, always power down module bay tower when removing/installing modules.
- Keep electrical cords in good working condition, and replace frayed and damaged cords.
- Do not replace detachable MAINS electrical supply cords with inadequately RATED electrical cords.
- 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.

### P.4.3 Cryogenics

- 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. Quantum Design recommends installing oxygen sensors where the MPMS 3 is located and with all other cryogenic systems.
- 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 and monitor for additional safety warnings and notices.

### P.4.4 Moving the MPMS 3

The MPMS 3 has four integrated caster wheels that allow the system to be easily rolled from one location to another. However, several steps must be taken in order to prepare the system for safe relocation. These detailed instructions can be found in Appendix A.3.6.

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## P.5 Disposal Information

The MPMS 3 is exempt with the requirements of:

- DIRECTIVE 2011/65/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 8 June 2011 on the restriction of the use of certain hazardous substances in electrical and electronic equipment (RoHS 2)

The MPMS 3 complies with the requirements of:

- DIRECTIVE 2012/19/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 4 July 2012 on waste electrical and electronic equipment (WEEE)

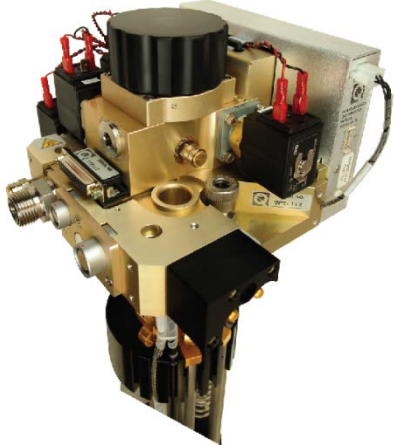

### P.5.1 RoHs Statement

RoHS Statement: The MPMS 3 is a Research and Development instrument that falls into Category 9 RoHs exemption.



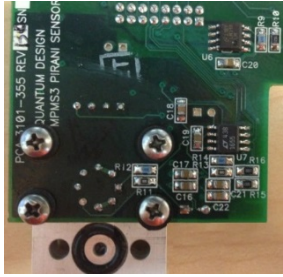
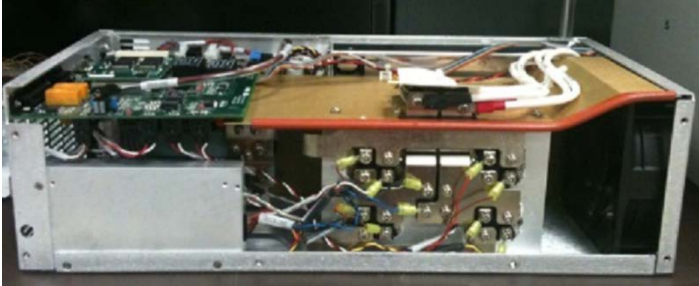

### P.5.2 WEEE Statement


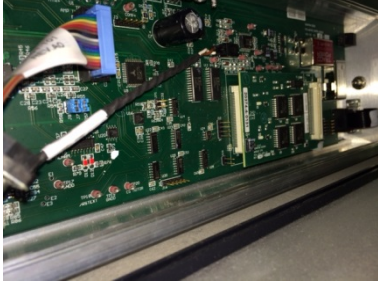
WEEE Statement: The MPMS 3 is WEEE compliant as Quantum Design uses recyclable materials in the fabrication of the equipment. Several components require special handling and processing and these components must be removed at time of decommissioning for proper handling before recycling/disposal. Contact Quantum Design for updated procedure/recommendations before disposal. A list of components to be removed before recycling or disposal is provided in Table P-1 below.

Table P-1. List of Components to be removed before Recycling or Disposal.

<i>Component Description / Location</i>	<i>Identifying Photograph/Diagram</i>
MPMS3 7T Probe Assembly (QD P/N: 4502-100)	
Power Drawer Assembly (QD P/N.: 4506-050)	



<p>MPMS3 backplane (QD P/N.: 3572-002)</p>	 A photograph of a green printed circuit board (PCB) backplane installed in a metal chassis. The board is populated with various electronic components and connectors, and is secured with screws.
<p>Gas Handling Module (QD P/N: 4501-040)</p>	 A close-up photograph of a green PCB assembly. It features a white connector, various electronic components, and a metal bracket. A label on the assembly reads "QUANTUM DESIGN GAS HANDLING MODULE MODEL 45040".
<p>Pirani Sensor and PCA Assembly (QD P/N.: 4501-071)</p>	 A photograph of a green PCB assembly. The board is populated with various electronic components, including resistors, capacitors, and integrated circuits. A label on the board reads "PQ101-355 REV1.0 SIN QUANTUM DESIGN MPMS3 PIRANI SENSOR".
<p>Hybrid Magnet Controller Assembly (QD P/N.: 4503-010-01)</p>	 A photograph of a Hybrid Magnet Controller Assembly. It shows a complex arrangement of electronic components, including a large green PCB, various connectors, and a metal chassis. The assembly is densely packed with components.
<p>Temperature Control Module Assembly (QD P/N: 4101-260)</p>	 A photograph of a Temperature Control Module Assembly. It shows a green PCB assembly with various electronic components, including a large integrated circuit, resistors, and capacitors. The assembly is housed in a metal chassis.

<p>SQUID Control Module Assembly (QD P/N: 4101-500)</p>	 A photograph of a SQUID Control Module Assembly. It shows a green printed circuit board (PCB) populated with various electronic components, including integrated circuits, capacitors, and resistors. The board is housed in a metal chassis. On the left side, there are several connectors, including two D-sub connectors and two BNC connectors. The text "CH 1" and "CH 2" is visible near the D-sub connectors.
<p>Motor Module Assembly (QD P/N: 4101-100)</p>	 A photograph of a Motor Module Assembly. It shows a green PCB with various electronic components, including a large integrated circuit, capacitors, and resistors. A multi-colored ribbon cable is connected to the board. The board is housed in a metal chassis. The text "CH 1" and "CH 2" is visible near the top of the board.



# DECLARATION OF CONFORMITY



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

## Manufacturer

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

## Product Name

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

## Standards to which Conformity is declared

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**LVD** EN 61010-1 2<sup>nd</sup> ed. : 2001,  
IEC 61010-1: 2001 (Second Edition)

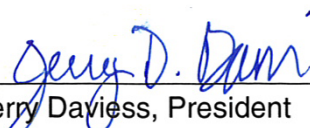
**EMC** EN 61326-1 :2006 Class A  
EN 55011: 2007,  
EN 55022: 2006,  
IEC 61000-4-2: 1995/A1: 1998/A2: 2000,  
IEC 61000-4-3: 2006,  
IEC 61000-4-4: 2004 + Corrigendum 1: 2006,  
IEC 61000-4-5: 2005  
FCC Part 15B, AS/NZS CISPR 11

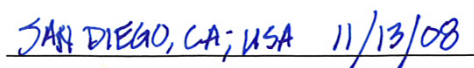
## Documentation Reference

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**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 Daviess, President

  
\_\_\_\_\_  
Place and Date

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

## Getting Started

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### 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 MPMS 3. 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.

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### 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 3 utilizes some analytic techniques employed by DC measurement scans. During the DC measurement the sample is moved slowly through the pick-up coils detecting the magnetic moment based on the scan time and amplitude of the measurement.

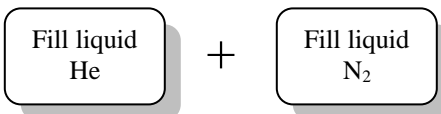
The MPMS 3 utilizes a superconducting magnet (a solenoid of superconducting wire) to subject samples to magnetic fields up to 7 Tesla (70 kOe). The SQUID detector and magnet must both be cooled with liquid helium. Liquid helium is also used to cool the sample chamber, providing temperature control of samples from 400 K down to 1.8 K. To help conserve liquid helium, the system is designed to use less costly liquid nitrogen to intercept heat bound for the helium tank. The MPMS 3 will only operate properly with both cryogenics in use: liquid helium and liquid nitrogen.

For details on hardware and measurement methods refer to Chapters 3 and 4, respectively

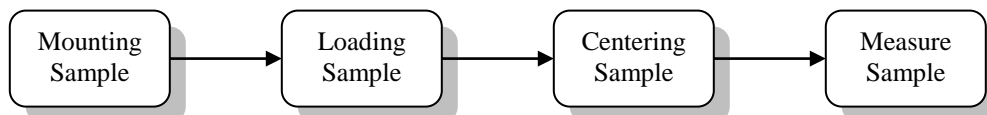
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## 1.3 Common Tasks

Before attempting a measurement make sure the system is filled with the necessary cryogenes:



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



### 1.3.1 Mounting Samples

In general, a few precautions should be taken when mounting a sample onto the sample holder to be measured on the MPMS 3:

- Make sure the sample will fit into the chamber. The inside diameter of the sample chamber is 9 mm. The sample dimensions should be well below this dimension to avoid rubbing of the sample through the sample chamber.
- For the most accurate magnetic moment measurements, measure the magnetic signature of the sample holder before mounting the sample. Once the sample is measured the background signal of the sample holder will need to be manually subtracted, point-by-point, from the sample measurement.
- Quartz and brass sample holders are supplied. In general, quartz sample holders should be used for measurement of small magnetic moments. The quartz holders are brittle and fragile, but have the smaller magnetic signature. For samples having a large moment, the brass sample holders are preferred and will have negligible magnetic background and may be easier to handle.
- Mount samples very securely to a sample holder to achieve accurate magnetic moment readings. (GE) 7031 varnish, or a glue that will withstand the temperature extremes and sample holder thermal contraction during the experiment is recommended for bonding the sample to the sample holder. In general, 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 will damage the adapter that mates the sample holder to the sample rod.
- 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.

- Use the supplied sample mounting station to place the sample at the proper location ( $66\pm 3\text{mm}$ ) on the sample holder as shown in Figure 1-1. The sample holder should be placed on the mounting station such that its base is aligned flush with the zero-point indicator.

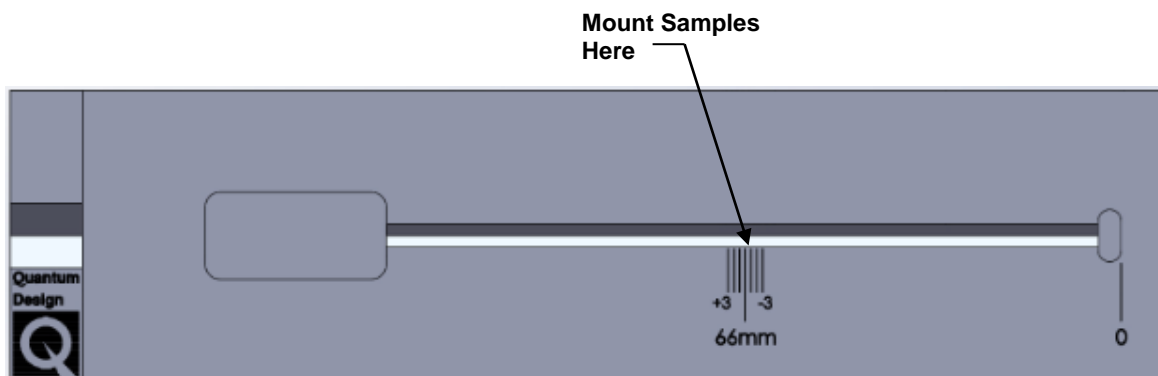


Figure 1-1. Sample mounting station.

**Note** While custom sample holders may also be used, it should be noted that the accuracy and reproducibility of magnetic moment readings may be compromised. The supplied brass and quartz holders are manufactured to ensure optimal geometry, and the MPMS 3 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.

### 1.3.1.1 OPTIONAL SAMPLE HOLDERS

Specialty sample holders may also be purchased from Quantum Design. Available options include straw and liquid sample holders. Guidelines for use of these specialty holders are outlined below.

#### STRAW SAMPLE HOLDER:

The hardware needed for using a straw sample holder include:

- Straw adaptor (QD Part # 4500-614)
- Straws (QD Part # 8000-001)

**NOTE** The straw sample holders are only intended for use with DC Scan mode. They should not be used with the VSM option.



#### WARNING!

Do not use straws at temperatures greater than 350 K. Straws will melt.

Like the quartz and brass sample holders, predetermined parameters of the straw holder-sample rod assembly allow use of the Auto-Tracking feature (see Section 1.4.3) during measurement. If the user chooses to use straws from other sources, it is possible that sample position may behave differently upon cooling and warming.

As shown in Figure 1-2, the straw fits snugly onto the adaptor, and a sample is mounted securely in the straw. The sample mounting station should be used to position the sample in the straw at  $66 \pm 3$  mm.



Figure 1-2. MPMS 3 sample rod, straw adaptor (yellow), and straw holder with sample.

**Note** The straw length should be modified so that it is roughly the same as the quartz or brass sample holders ( $5.82 \pm 0.01$  in).

A basic procedure for using straw sample holders is provided below. Additional guidelines for mounting thin film, gelatin powder capsule, and single crystal samples in straw holders are given in *Application Note 1500-018: Using Straw Sample Holders with DC Scan Mode in MPMS 3*.

1. Weigh and measure the sample. After you insert the sample into the sample chamber, you can select Sample > Properties... from the menu bar to define the sample's mass, in milligrams, and its diameter and length, in millimeters. Here you can also specify the type of sample holder used.
2. Use two short sections of straw with thru cuts, and slide them into the outer straw to sandwich the sample in the middle. The straw segments should be near identical in shape and length to ensure the background is uniform, and thus contributes little to the overall signal. Use the sample mounting station to make sure that the sample is positioned at  $66 \pm 3$  mm.

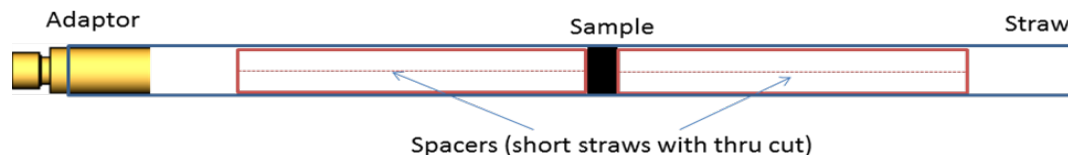


Figure 1-3. Correctly positioned sample in straw holder.

3. Alternatively, the sample can be placed inside of a small straw segment as shown below.
  - a. To mount your sample in this manner, use phenolic tweezers to place the sample inside the small straw segment.
  - b. Hold the straw segment so that its two open ends are vertical.
  - c. Place the straw segment inside the drinking straw, and move the segment until it is  $66 \pm 3$  mm from the end of the straw. Verify that the walls of the straw obstruct the open ends of the segment.

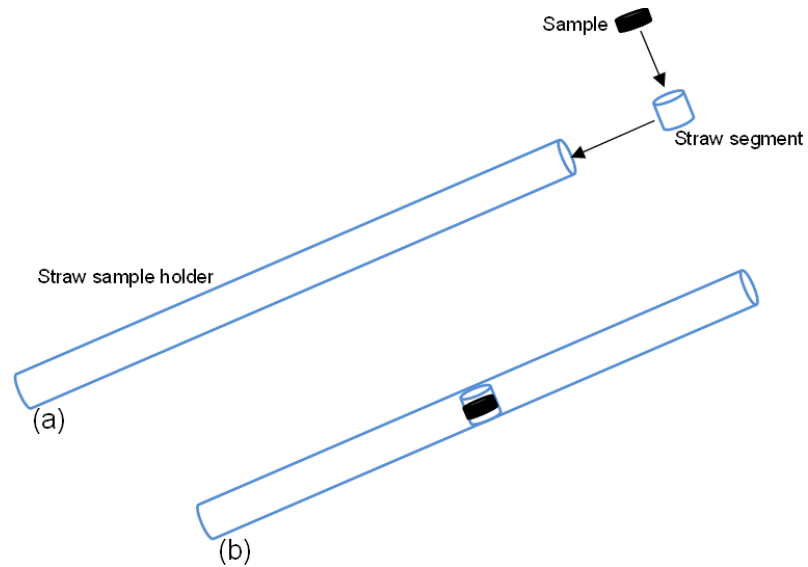


Figure 1-4. Using a small straw segment to position a sample in the straw holder.

4. To attach the straw to the sample rod adaptor, wrap a sufficient amount of Kapton tape around the adaptor so that the drinking straw will fit snugly over it.
5. Place the end of the drinking straw over the tape on the adaptor.
6. Place a small piece of tape over the exposed end of the drinking straw. This extra piece of tape prevents a loose sample from falling into the sample chamber.

**SEALED SAMPLE HOLDER:**

Sealed Teflon sample holders are also available from Quantum Design. The sealed sample holders can be used to measure solid and powder samples. Liquid samples may also be measured if grease or Teflon tape is applied to the fine threads of the Teflon sample bucket. However, it is recommended to test the seal in another vacuum space prior to measuring in the MPMS 3. Due to its high background signal the sealed sample holder should not be used to measure weakly magnetic samples.

Necessary hardware:

- Sealed Sample Rod Assembly (QD part # 4084-266)
- Adaptor (QD part # 4500-654)

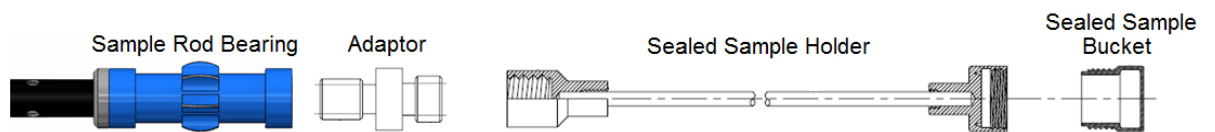


Figure 1-5. MPMS 3 sample rod, adaptor, and sealed sample holder.

The body of the sealed sample holder is a carbon fiber rod. The Teflon sample bucket threads onto the sample holder rod. The adaptor is mated with the Teflon female joint at the top of the sample holder. The background signal of the sample holder should be measured prior to measurement of the sample.



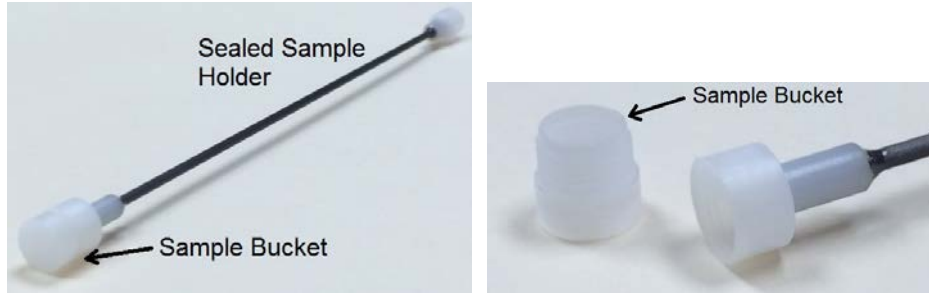


Figure 1-6. Sealed sample holder and sample bucket shown in detail.

### 1.3.2 Loading and Unloading Samples

1. Attach a 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 1-7. Sample holder mounting.

2. Select the software menu item “Sample > Install/Remove...”, select the “Change Sample” button from the toolbar or the change button in the sequence control window.

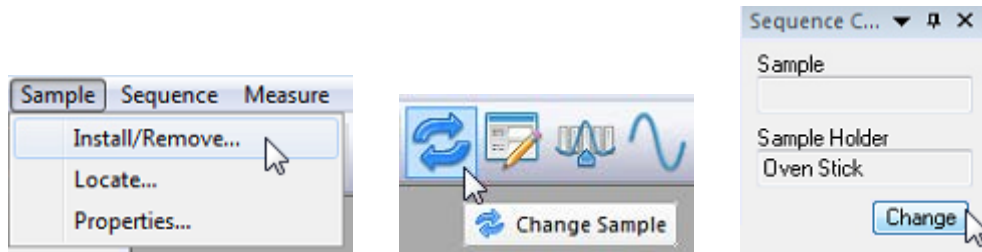


Figure 1-8. Sample installation menu, toolbar button, and sequence control pane.

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.

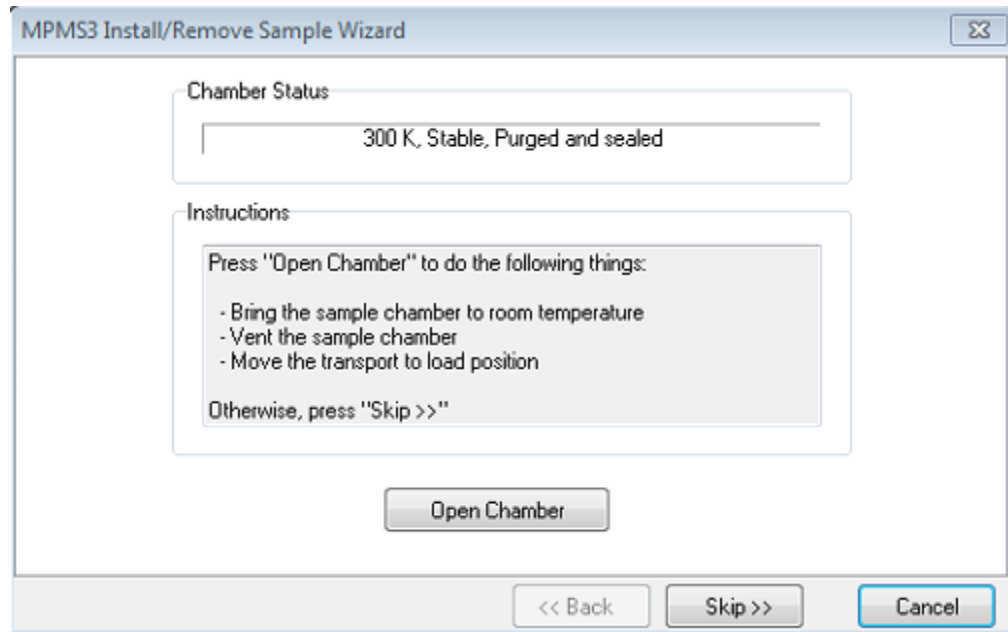


Figure 1-9. Sample installation wizard.

3. Follow the instructions in the wizard.
4. After specifying a data file for saving measurement information, the “Sample Properties” window will appear and you will be prompted to enter optional information about the sample and sample holder.

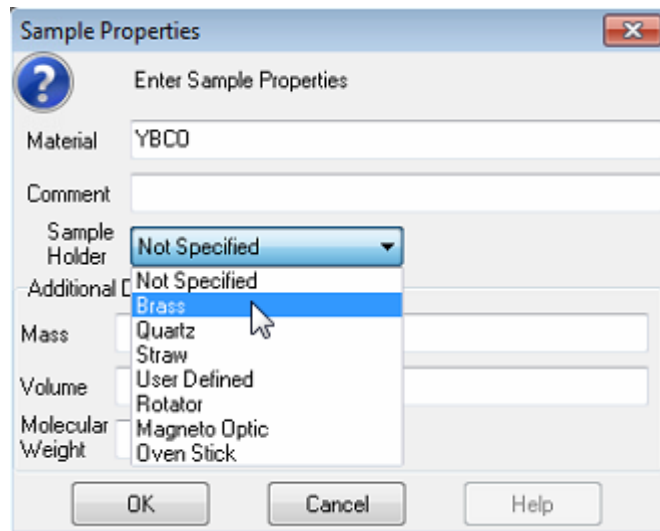



Figure 1-10. Sample Properties Window

Selecting one of the Quantum Design sample holders (brass, quartz, straw) for your measurement will enable the advanced feature called Autotracking. In this advanced feature the system automatically tracks the center of the sample during cooling and warming, based on the predetermined thermal expansion properties of the QD sample rod-sample holder assembly.

**NOTE** Sample properties entered are used for informational purposes only. Magnetic moment is always measured in either emu or A·m<sup>2</sup>. Refer to Section 2.2.1 for information on changing the measurement units.

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 the “Eject” button, , on the front instrument bezel. The chamber vents continuously with helium and the sample transport moves to the top position.
3. Remove the cap from the sample transport.

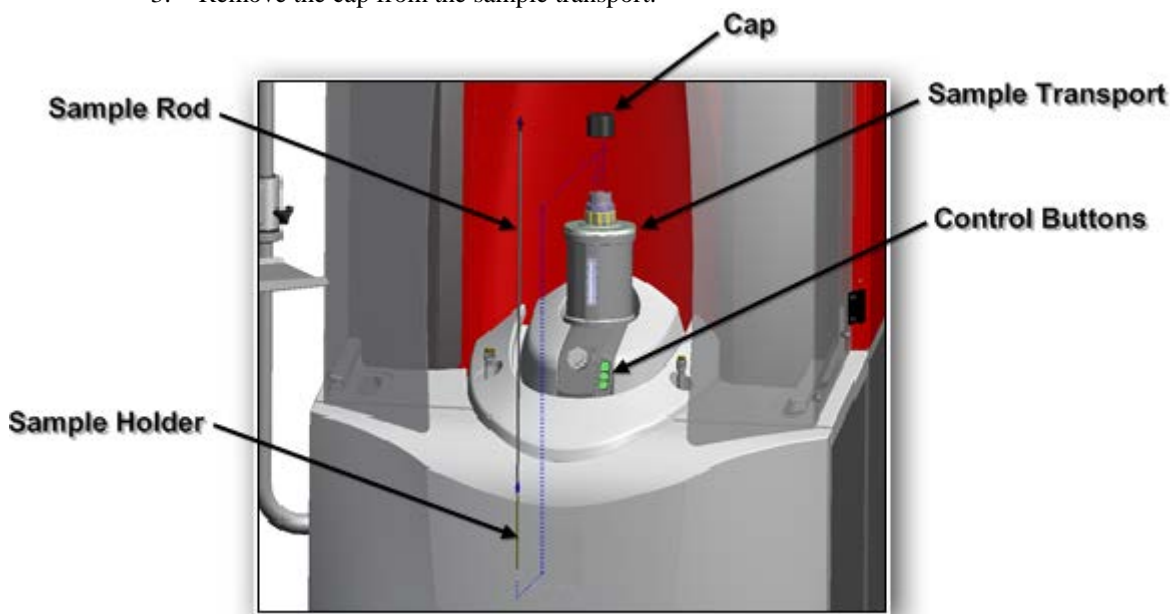



Figure 1-11. Sample rod installation.


5. Remove the installed sample rod. Insert a new sample rod into the chamber if desired.
6. Replace the cap and press the “load”  button on the instrument bezel.

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



**WARNING!**

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

 **WARNING!**

Before installing the sample transport, check that the O-ring on the sample space manifold is in place (Figure 1-11) and is free of debris.

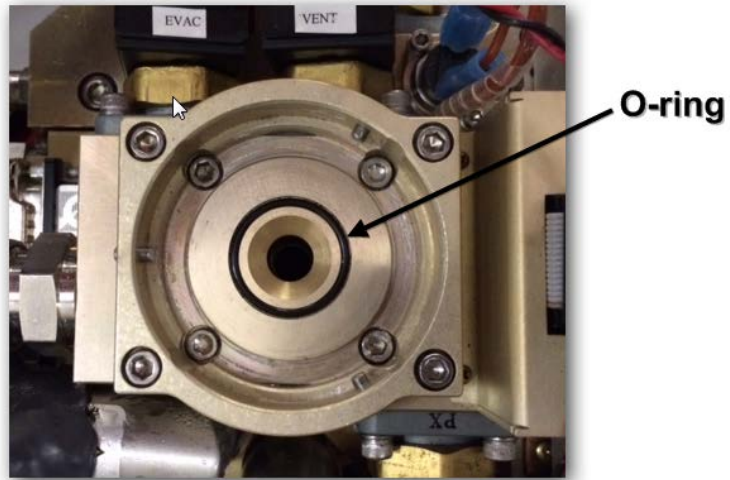



Figure 1-12. Sample space manifold O-ring.

 **WARNING!**

Never insert the sample rod into the sample chamber if the O-ring on the top of the sample transport is missing (see Figure 1-13). The O-ring prevents air from pumping into the chamber. Air pumped into the chamber can damage the vacuum pump and freeze the sample.

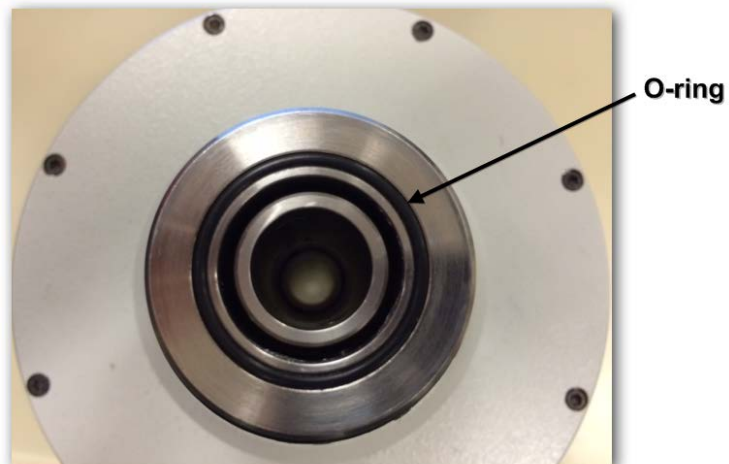


Figure 1-13. Sample transport O-ring.

## 1.4 Centering Samples

Before initializing a single measurement or a sequence, the sample must be centered within the detector coils. During a centering scan, the MPMS 3 scans the entire length of the sample transport's vertical travel path. 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 above in Section 1.3.1 "Mounting Samples".

There are two different ways to enter the sample offset: **Scan for Sample Offset** and **Enter Offset Manually**. The Install/Remove Sample Wizard will scan for sample offset or let you specify the offset value. If you are not using the Install/Remove wizard, select **Sample > Locate** or select the "Locate" button from the toolbar.

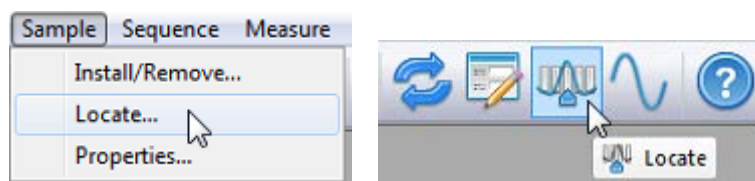


Figure 1-14. Locate menu and toolbar button.

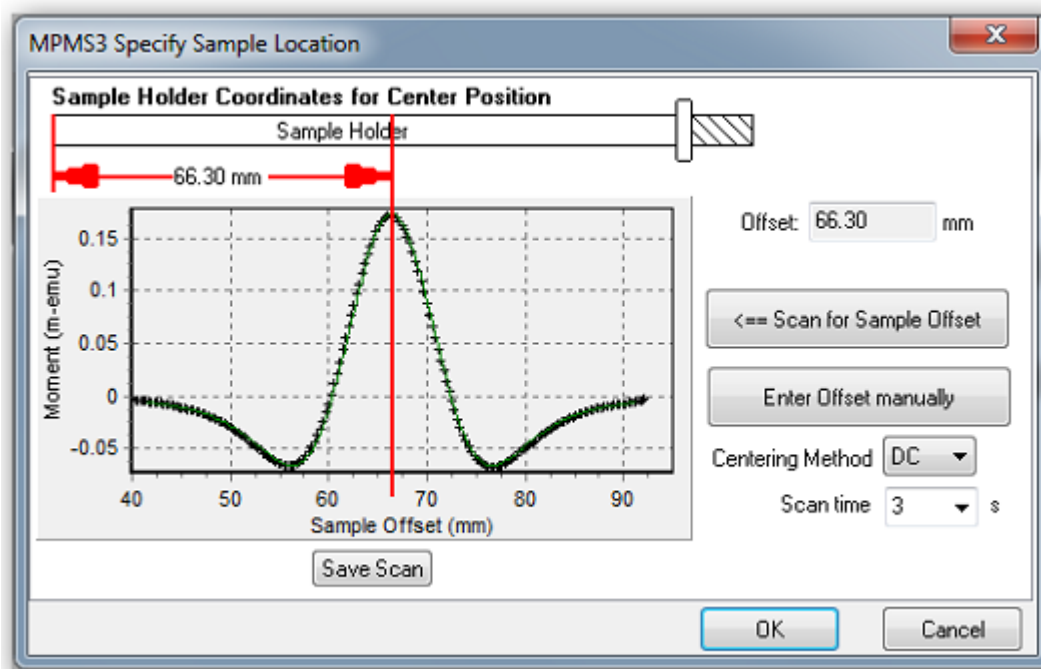


Figure 1-15. Sample centering scan.

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 mm. After a successful centering scan or manual entry of the sample offset, the motor armature position should be roughly centered in the window of the sample transport.

1. The most accurate technique is to **Scan for Sample Offset**. The software moves the sample through the detection coils and analyzes the coil response to locate the sample. The MPMS 3 initializes the sample transport by first performing a “touchdown” to the lower-travel-limit. The initialization position places the sample far enough below the pickup coils that the SQUID does not detect the sample moment. Centering scan parameters are factory defined, with the exception of Scan time, which can be set from 1-15 seconds. The default centering scan time is 4 seconds. However, for larger moment samples, slower scans should be used to avoid software errors in reconstruction of DC waveforms. Alternatively, to perform a good centering scan on samples with small moments, you may need to induce a magnetic moment  $> 1 \times 10^{-7}$  emu in your sample by applying a magnetic field, or by changing the temperature.

Figure 1-15(a) illustrates the sample moving upward through the detection coils. Figure 1-15(b) illustrates MPMS 3 MultiVu measuring the SQUID response while the sample moves through the coils. Figure 1-15(c) plots the SQUID response against the scan length; the output in Figure 1-15(c) indicates that the sample is mounted too high and must be lowered. In Figure 1-15(d), the scan begins one-half scan length below the center coils.

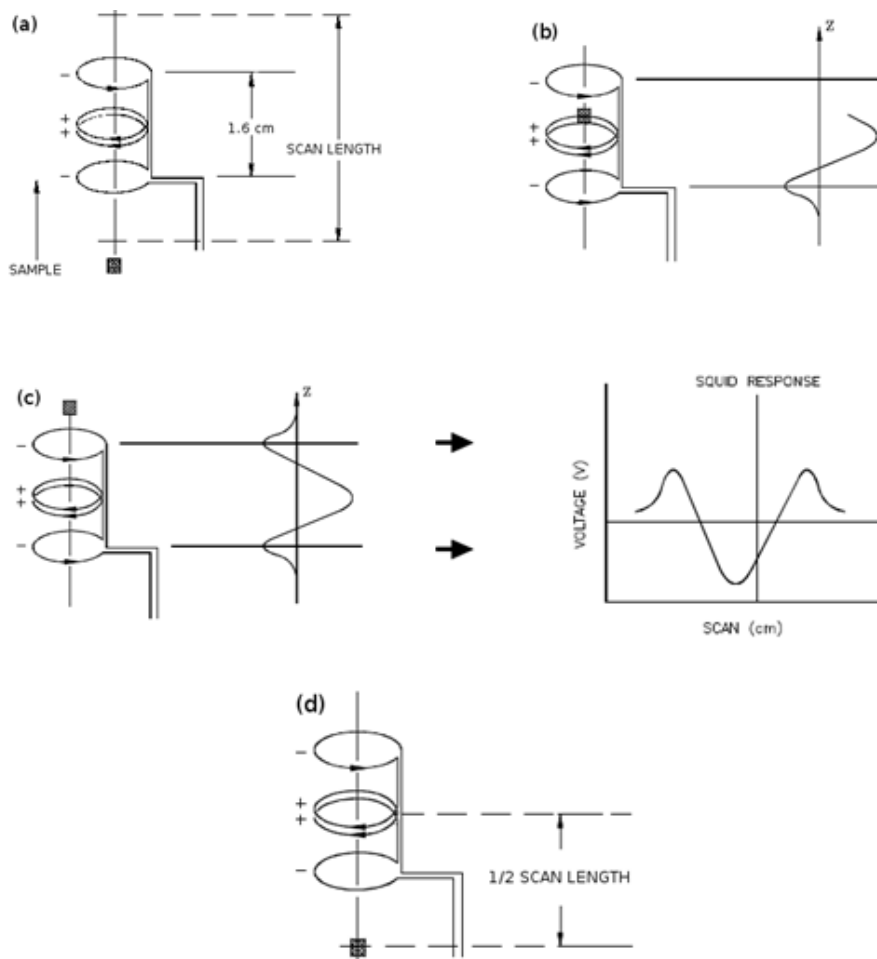


Figure 1-16 DC centering scan and measured SQUID voltage response.

2. If your sample has a very weak magnetic response and inducing a magnetic moment is undesirable or impossible, scanning the sample may not work. Additionally, 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 using the sample mounting station before loading the sample and select **Enter Offset Manually**. This is a necessary step to ensure that samples are properly centered during measurements.

Sample offset is the distance measured from the bottom end of the sample holder to the (magnetic) center of the sample. When mounting your sample, use the sample mounting station to ensure that the magnetic center of your sample falls within the center of the gradiometer coils, at  $66 \pm 3$  mm.

### 1.4.1 Scan the Sample

By scanning the sample before measurements, the user can find the center of the sample in between the pick-up coils.

1. Click “Scan for Sample Offset.” The instrument smoothly moves the sample through the detection coils. It may scan the sample several times to achieve optimum signal. The user may adjust the scan time that it takes the system to center a sample.
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.

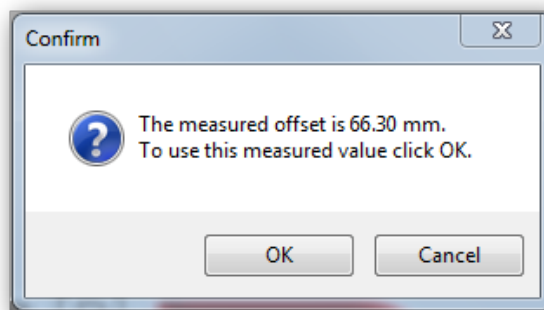


Figure 1-17. Sample offset confirmation.

### 1.4.2 Enter Sample Offset Manually

Samples with weak magnetic response might need to be centered manually. To do so:

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 should be  $66 \pm 3$  mm. For accurate measurements, it is important to enter the offset as close as possible to the true value. If your sample offset differs from this range, the sample should be repositioned on the sample holder.

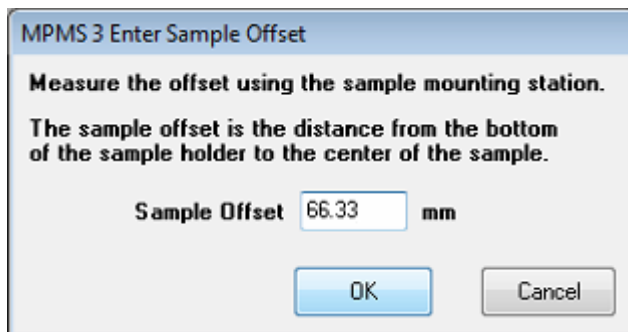


Figure 1-18. Manual Sample Offset

4. Click “OK.”

### 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 (see Figure 1-20).

- 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 first be specified in the “Sample Properties” dialog. Auto-Tracking will generally give the best results and is the preferred setting in the MPMS 3.
  - You may also disable automatic centering with **No 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 the “Setup” tab in the MPMS 3 Measurement window.

### 1.4.4 Measuring Samples

With DC measurements the sample is scanned up and down at constant speed and the SQUID signal is obtained as a function of position and time. The up and down scans are subtracted in position space, giving an approximation of the system drift over the time of the measurement. Linear drift is then subtracted from the SQUID signal as a function of time, and the raw data is fitted to the dipole response function in position-space. The fitting algorithm assumes that the sample magnetic moment approximates a dipole magnetic moment, with sign and value remaining constant during measurement. DC Fixed Center and DC Free Center moments are calculated for each point, based on the dipole response function. As the names suggest, for Fixed Center the center position has been fixed for the fitting routine, and with Free Center, the center position is a free parameter. The auto-tracking feature mentioned above is particularly useful for the DC Moment Fixed Center. The auto-tracking center acts as the seed center position for the DC scan. For more on DC measurement theory, refer to Chapter 4.



### 1.4.4.1 IMMEDIATE MEASUREMENTS

Immediate measurements refer to the magnetic moment of a sample at a specific temperature or magnetic field.

- Initiate sample measurement with the menu item **Measure > Measure...** or by clicking the “Measure” toolbar button. Either method will open the Measurement window (Figure 1-20).

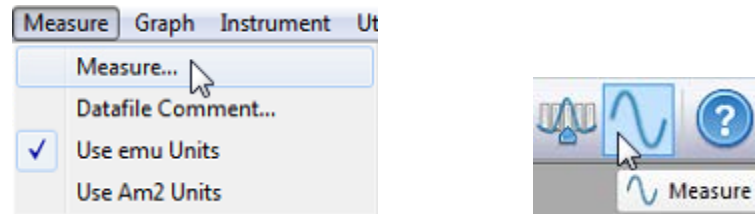


Figure 1-19. Selecting "Measure" from either the pull-down menu or the toolbar button

Base system tabs include **Setup**, **DC**, and **Graph**. *Additional tabs may be present for installed options such as VSM or AC.* Descriptions of the measurement tabs and parameters are given below.

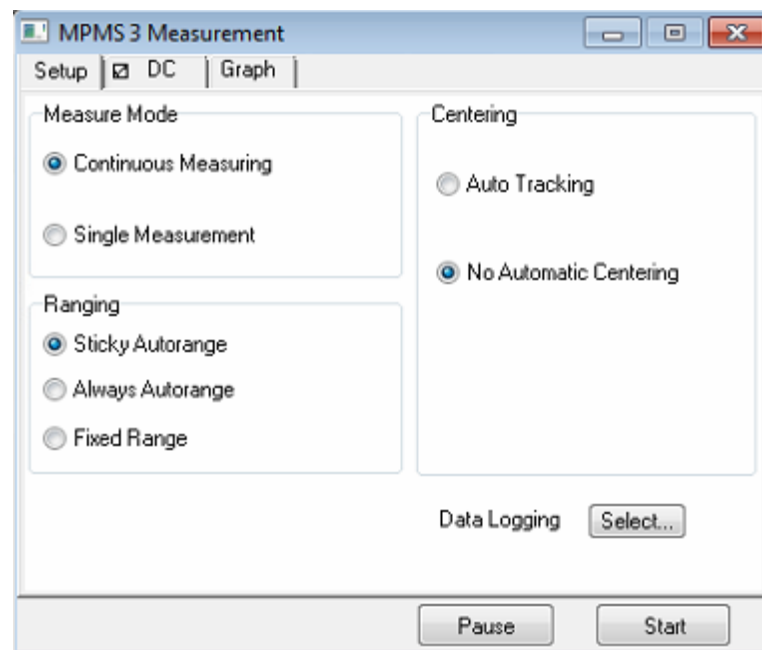


Figure 1-20. MPMS 3 Measurement window.

- **Setup Tab**
  - Specify “Continuous Measuring” for a continuous stream of data, or “Single Measurement” to collect a single data point.
  - Specify the SQUID “Ranging” as **Sticky Autorange** (default), **Always Autorange**, or **Fixed Range**. Sticky Autorange selects the best range for each point while minimizing re-ranging between individual data points. Always Autorange acquires each data point with the best possible resolution even if the measurement will take longer due to the increased range changing operations. Fixed Range uses the SQUID range specified by the user.

For additional details regarding SQUID ranging, refer to Appendix B: *Advanced Software Options*.

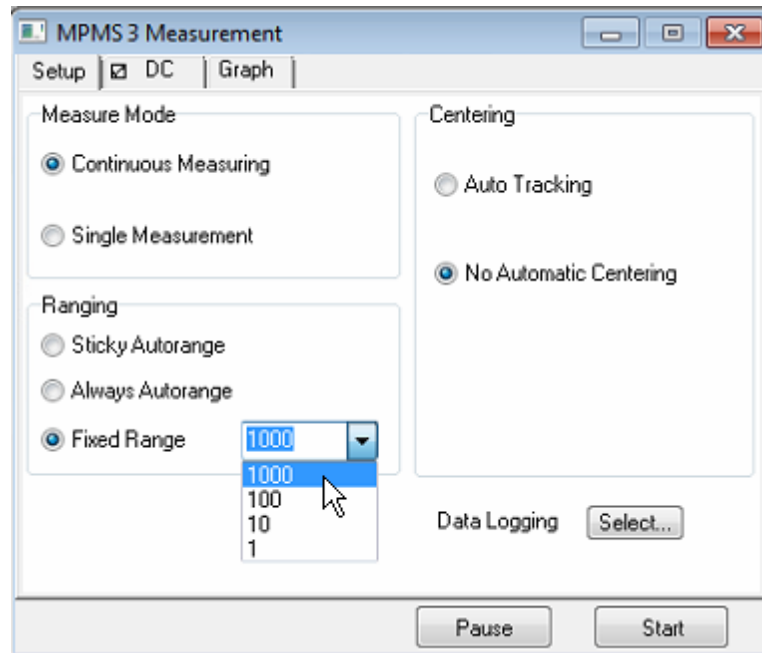


Figure 1-21. Selecting a fixed SQUID range.

- Specify which automatic centering method is used to keep the sample centered during measurement:
  - **Auto Tracking** – Automatically tracks the sample during cooling and warming based on known parameters of the sample rod-sample holder assembly. In order to select this option, a Quantum Design supported sample holder must first be selected under Sample Properties. Auto Tracking is disabled when using custom sample holders.
  - **No Automatic Centering** – Disables automatic centering
- “Data Logging” (Figure 1-23) allows you to select a \*.qmap file to log additional diagnostic information such as gas handling, temperature control, and magnet control data. The data will be stored in the measurement data file. An example of diagnostic data logging is shown in Figure 1-24. This differs from the MutliVu log which can be run in the background in addition to your measurement data log. For more information about running a diagnostic log in the background, refer to Appendix B.3.

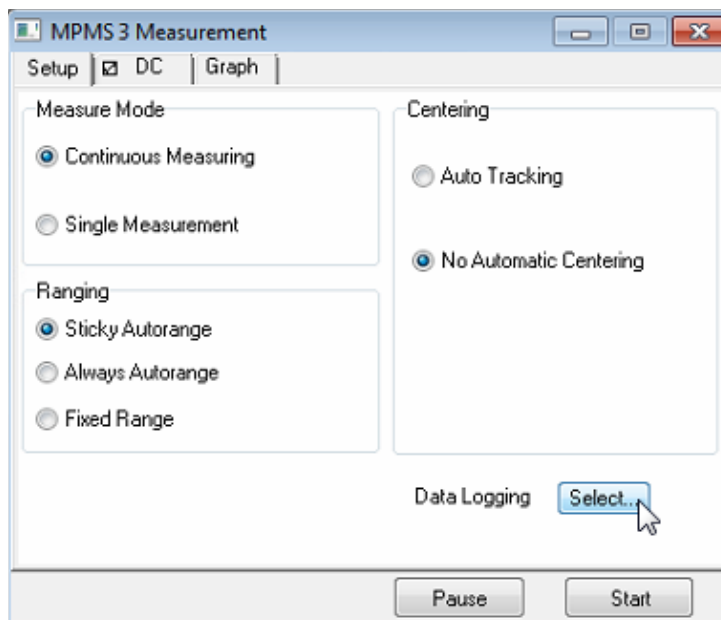


Figure 1-22. Data logging option.

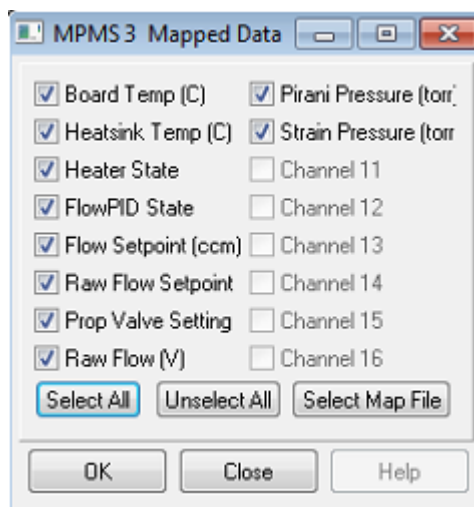


Figure 1-23. Example of data selections to be logged.

▪ **DC Tab**

- **Scan Length** – Specify the total scan length (in mm). The value entered is the total length of the scan, which will be centered about the expected center position of the sample. Scan lengths can be set between 10 mm and 60 mm (default: 30 mm).
- **Scans per Meas.** – The total number of scans (up + down) to be acquired before the data is analyzed to calculate the sample moment.
- **Scan Time** – Time (in seconds) for each individual (up or down) scan. In addition to the values available from the drop down menu, non-integer values may be entered as well (optimal scan times depend on environmental conditions). The default scan time is 4 seconds. However, scan times may need to be adjusted based on the magnitude of your sample moment. For samples with large moments, or time dependent magnetization, faster scans may be required.

**NOTE** Additional measurement considerations for the above DC parameters may be found in the following Application Notes:

- 1500-229, *DC Scan and High Moment Samples*
- 1500-018, *Using Straw Sample Holder with DC Scan Mode.*

- **Save Raw Data** – Save raw scan data for each data point (into a separate \*.raw file) for later analysis or post processing. The raw data file will be saved in the same folder as the measurement data file, under the same name as the data file, but with the \*.raw file extension.

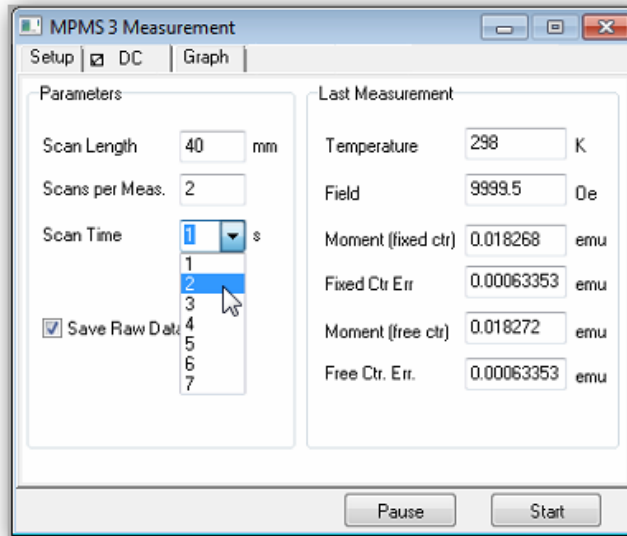


Figure 1-24. DC measurement parameters.

- **Graph Tab** – Shows the up and down scans (black) of the last DC measurement and the mathematical fit to the raw data (green).

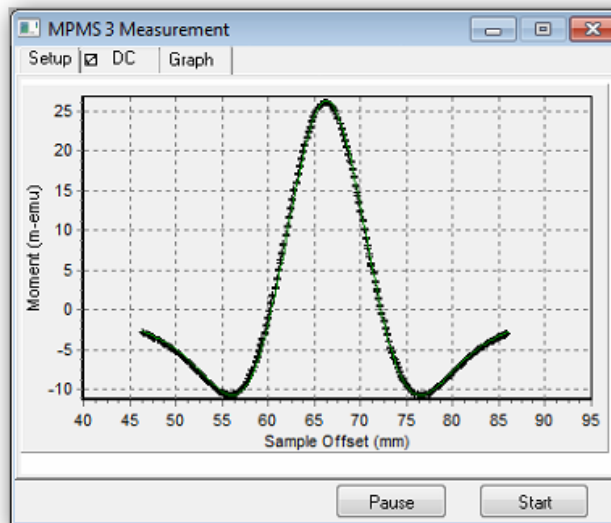


Figure 1-25. Calculated moment of the up and down scans (black) for the last DC measurement, along with the mathematical fit to the data (green).

Before clicking “Start” to begin the immediate measurement, temperature and magnetic field can be set by selecting **Instrument > Temperature...** (or **Instrument > Field...**), or by clicking on either the temperature or field status bars (see Figure 1-27). Both the system temperature and field control windows (Figure 1-26) allow the user to choose a set point and rate, as well as mode of approach. Additional details regarding the status bar may be found in Section 2.2.5. Approach modes are discussed in Section 2.3.5.2.

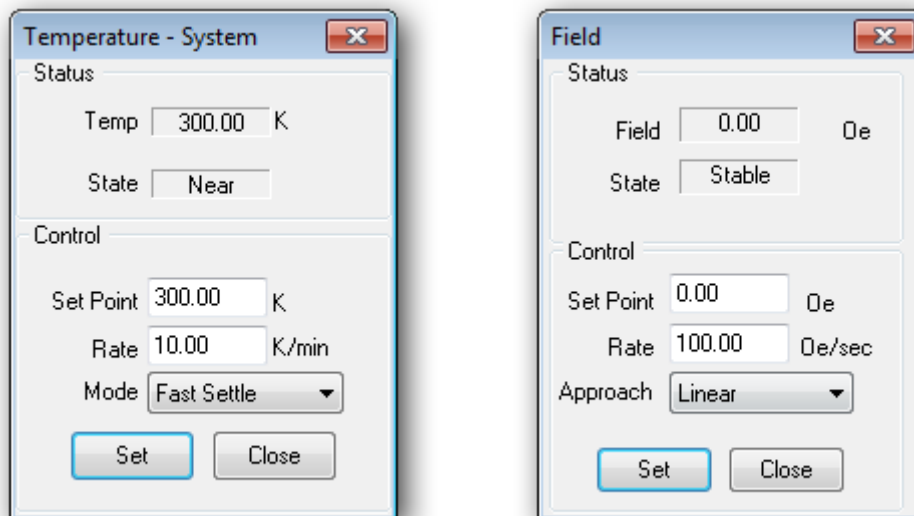


Figure 1-26. System temperature and magnetic field control windows.

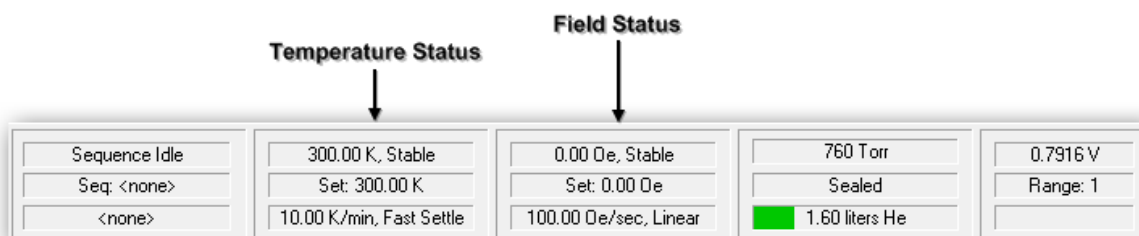


Figure 1-27. MPMS 3 MultiVu status bar.

For temperature, the stability criteria are:  $\pm 0.5\text{K}$  for  $T > 100\text{ K}$ , and  $\pm 0.5\%$  for  $T < 100\text{K}$ . Field stability is declared after the QuickSwitch heater has been turned off and the QuickSwitch becomes superconducting.

#### 1.4.4.2 SEQUENCE (AUTOMATED) MEASUREMENTS

Automated measurements are controlled through sequence files, where operation commands are input by the user in a desired order. In addition to measurement commands, several other operations are also available through sequence measurements, all of which are summarized in Chapter 2.

### 1.4.4.3 SUMMARY OF DC SCAN PARAMETERS

Tables 1-1 and 1-2 below summarize the accepted DC and SQUID scan parameters for both DC centering and DC measurement scans. Default values are indicated when applicable.

Table 1-1. DC Scan Parameters

PARAMETER	ACCEPTED VALUES		DEFINITION
	CENTERING SCAN	MEASUREMENT SCAN	
Scan Length	60 mm	10-60 mm (1- 6 cm) Default: 30 mm	Length of sample transport's vertical travel path that is scanned. Sample is centered when it is in the middle of the scan length.
Scans Per Meas.	1	1-100	Number of scans (up + down) to be completed before data is analyzed and sample moment is calculated. This value does not account for transport motion as a result of SQUID ranging.
Data Points	~200	~200	Individual voltage readings plotting response curve in data file.
Scan time	1-15 s Default: 4 s	1-15 s Default: 4 s	Time in second for each individual up or down scan. Non-integer values within the 1-15 second range may also be entered.
Centering	N/A	Autotracking	Sample location is adjusted automatically based upon the known thermal response of the QD sample holders and sample rod.
		No Autocentering	Disables automatic centering.


Table 1-2. SQUID Parameters

PARAMETER	ACCEPTED VALUES		DEFINITION
	CENTERING SCAN	MEASUREMENT SCAN	
Ranging	Default: Sticky Autorange  (Not settable by user.)	Sticky Autorange  Always Autorange  Fixed Range (1000, 100, 10, or 1)	SQUID range is a value of sensitivity (see Appendix B), with 1 being the most sensitive.  Sticky Autorange (default) changes range only if the signal falls outside of a specified percentage of the full range.  Always Autorange dynamically adjusts the range for maximum signal, for every single measurement.  Fixed Autorange will keep the SQUID at the selected range; however, if the signal exceeds the maximum of the selected range, no data points will be collected.


For additional information regarding DC measurement theory and applications, including sample and measurement considerations, refer to Chapter 4 of this user's manual.

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## 1.5 Filling the Helium and Nitrogen Tanks

	<p><b>WARNING!</b></p> <p>Cryogenics 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 back stream out of fill ports when removing caps/plugs/transfer lines.</p>
---	---


**WARNING!**



To guard against these dangers, wear eye protection and protective clothing such as gloves and covered shoes when working with these cryogenes. 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.**

**WARNING!**



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 3 usually requires nitrogen about as often as it requires helium. It is recommended to fill the nitrogen and helium tanks at the same time. Monitor the nitrogen level meter, as the presence of nitrogen is critical for low temperature operation and minimizing consumption of helium. As older MPMS 3 systems do not have an integrated nitrogen level meter, it is very important to maintain a regular LN2 filling schedule. Generally liquid 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 shut down 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.

	<b>WARNING!</b>
<p>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.)</p>	

1. Connect the hose to the nitrogen storage dewar.

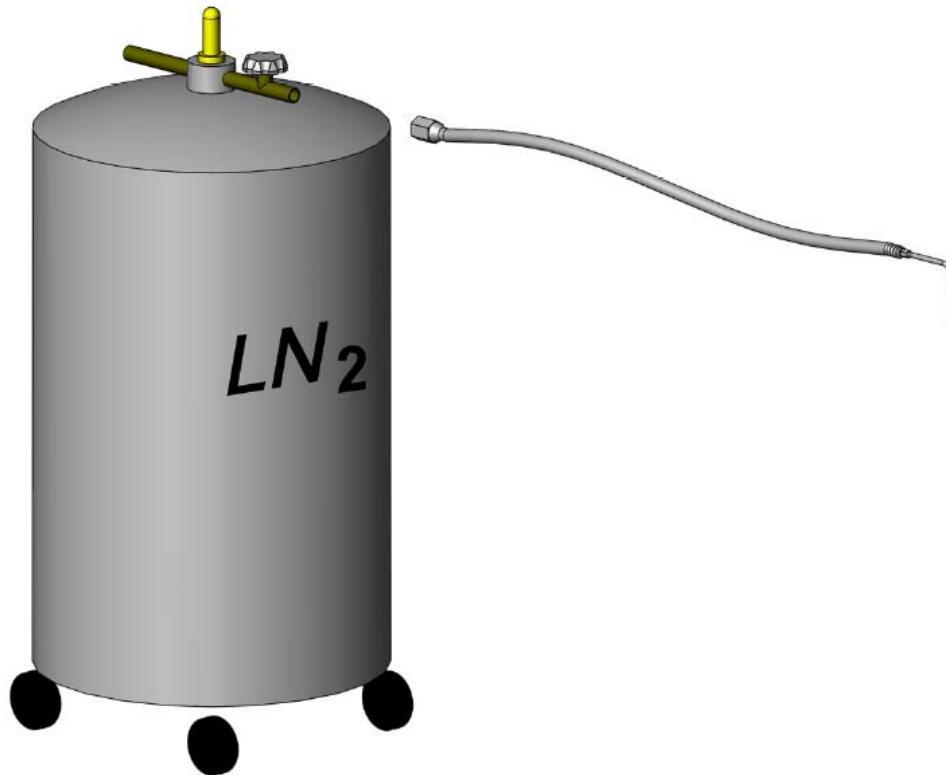


Figure 1-28. Liquid nitrogen transfer tank (only use low pressure containers  $p < 22$ psi, 1.5 bar).

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

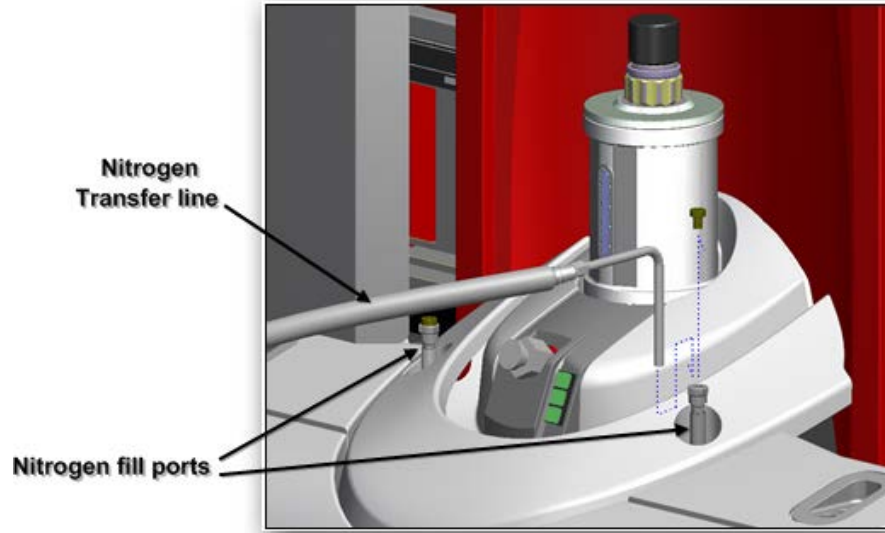





Figure 1-29. Filling liquid nitrogen.

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


	<b>WARNING!</b>
	<b>CAUTION:</b> 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 3 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.

	<b>WARNING!</b>
	<b>CAUTION:</b> Cold nitrogen gas/liquid might backstream out of the open port.


	<b>WARNING!</b>
	<b>SERVICE NOTE:</b> 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



**WARNING!**

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




**WARNING!**

Wear protective safety gear, such as gloves and goggles.

Start the helium fill utility:

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

OR Press the “fill”  button

- The helium fill status and graph will be displayed. If this is an initial cool down (or if the system has run dry) the software will ask whether this is an initial cool down or not. Selecting “Yes” will start the cool down wizard instead of the normal Helium fill (see Appendix A.4).

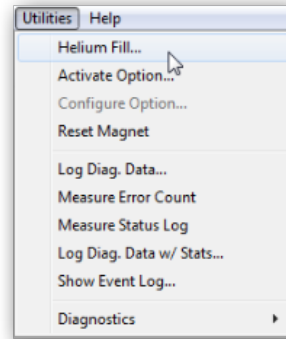


Figure 1-30. Utilities menu.

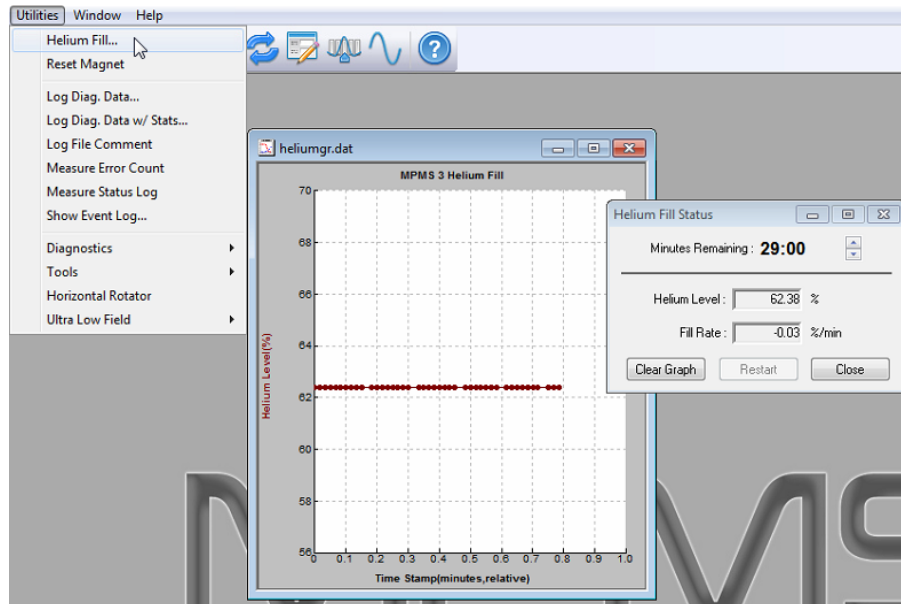


Figure 1-31. Helium fill utility.

- Install the Helium transfer line extension and insert the Helium 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 Helium 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.

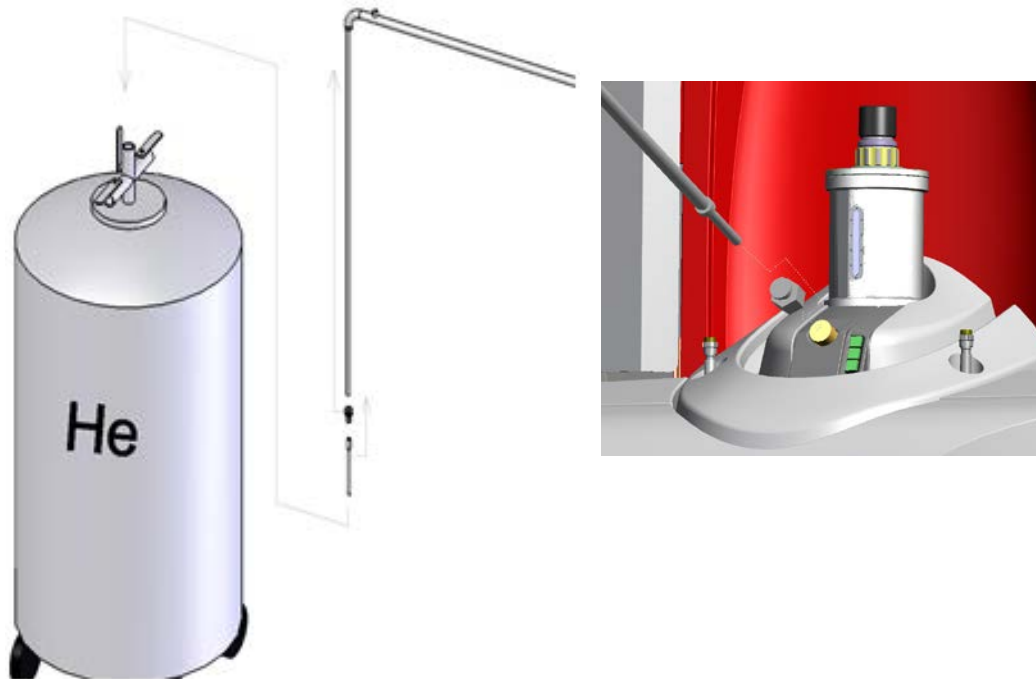



Figure 1-32. Liquid helium storage dewar, and helium transfer line insertion into fill port.

- Open the helium fill port on the MPMS 3 and rapidly insert the transfer line (Figure 1-32), then screw the fitting tight.

	<b>WARNING!</b>
	<b>CAUTION:</b> Cold helium might back stream out of the open fill port.

- Monitor the transfer as follows:

- a. Lower the Helium 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.
- o 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 MPMS 3 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 Helium 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.
  - o In order to determine the level of helium remaining in your storage dewar, use the Taconis tube (i.e., “thumper” or “dip-stick”) that was provided with your MPMS3. Instructions for using the Taconis tube may be found on the Quantum Design website in *Application Note 1014-404: Using the Taconis Tube*

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## 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 1-3. Button Functions




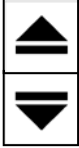
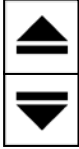
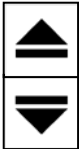



Button	Name	Used to...	Function when Pressed
	Eject	eject samples and open chamber	Vents sample chamber continuously with helium and moves sample transport to top of travel. Disabled when chamber is not at room temperature and when measurement is in process.
	Load	close chamber	Purges sample chamber 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 1-4. 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 > Show 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



# Software

## 2.1 Overview

**In this chapter** we describe MultiVu, the Windows™ software that coordinates the operation of the MPMS 3 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.

MultiVu may be freely installed on additional computers. To install MultiVu, simply run the “MPMS 3 Setup.exe” program and follow the instructions provided within the setup program. To install MultiVu on a computer that is not connected to your MPMS 3 system, you would select “Simulation Mode” from the popup list in the “Select Components” step – depending on the options that are present in your system, you might want to enable some of the option installers as well (see Figure 2-1).

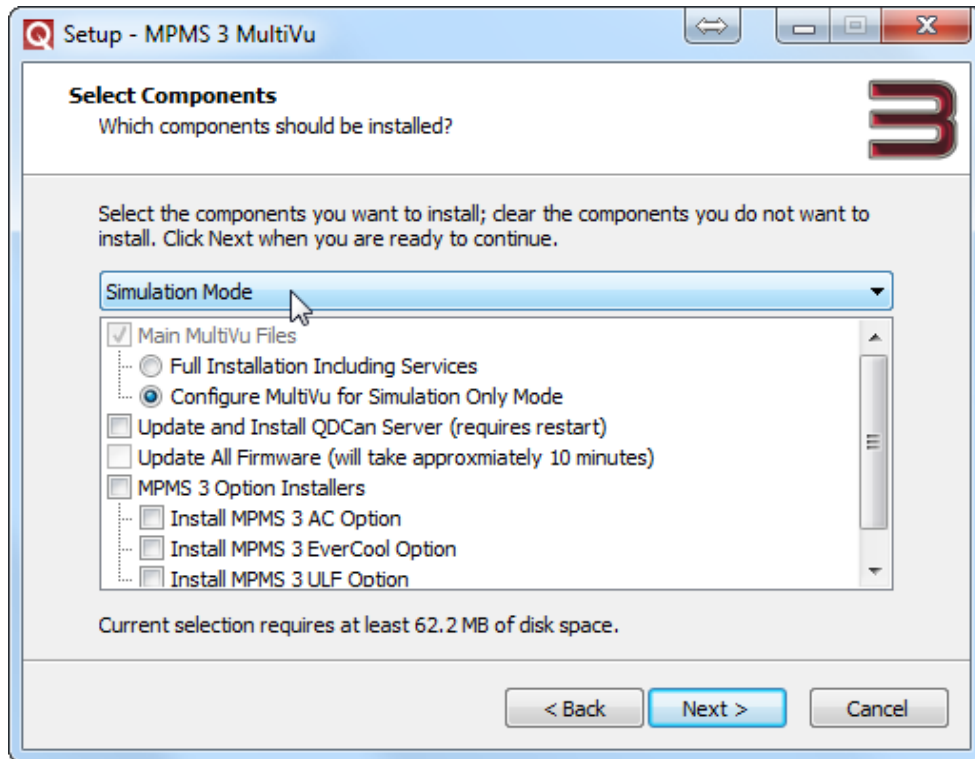


Figure 2-1. Components selection dialog in the MPMS 3 setup.



Once installed, use the desktop icon for running MultiVu in simulation mode, which works without MPMS 3 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 MPMS 3 computer.

To start the MPMS 3 MultiVu do one of the following: (a) select and double click the desktop icon or (b) open the Windows Start menu and then locate and select the MPMS 3 MultiVu option. The MPMS 3 MultiVu option may be located in the **Programs > Quantum Design** folder.



Figure 2-2. MPMS 3 MultiVu icon.

Wait for the MPMS 3 MultiVu to start up. The MPMS 3 MultiVu interface opens, and in the center of the interface, the startup progress dialog box appears. This dialog box identifies each task the system performs as part of the start-up. The dialog box disappears as soon as start-up is complete.



Figure 2-3. System startup progress dialog.

When the MultiVu software is not running, the MPMS 3 goes into a hibernation state to prevent equipment damage. Most users will leave MultiVu running on the MPMS 3 computer at all times. MultiVu has a control to place the MPMS 3 in standby 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. Table 2-1 lists the directories and descriptions of files associated with the MPMS 3 MultiVu Software.

Table 2-1. MPMS 3 MultiVu Directories and Files

DIRECTORY	FILES	DESCRIPTION	MANUAL REFERENCE
C:\QdSquidVsm\		Root directory containing all files related to MPMS 3 MultiVu	
C:\QdSquidVsm\Data		Default directory to store all measurement data	
C:\QdSquidVsm\Macros		Directory for basic scripts	2.3.2
C:\QdSquidVsm\MultiVu	SquidVsm.exe	MPMS 3 MultiVu program file	
C:\QdSquidVsm\MultiVu	*.dll	Libraries required for MPMS 3 MultiVu (list of files depends on installed options)	
C:\QdSquidVsm\MultiVu	*.chm	On-line manuals	
C:\QdSquidVsm\MultiVu	Event.log	Log file containing diagnostic information for trouble shooting	
C:\QdSquidVsm\Qmaps	*.qmap	Definition files for additional data logging	Appendix B
C:\QdSquidVsm\Sequence		Default directory to store sequences	2.1.2
C:\QdSquidVsm\SVsm\Calibration	Coil-....cfg	System-specific calibrations	
C:\QdSquidVsm\SVsm\LogFiles	ScanData.dat	Result of last location scan	
C:\QdSquidVsm\SVsm\LogFiles	VsmLog.txt	Log file containing diagnostic information for debugging related to the measurement DLL	

<b>DIRECTORY</b>	<b>FILES</b>	<b>DESCRIPTION</b>	<b>MANUAL REFERENCE</b>
C:\QdSquidVsm\SVsm\System	SVsmMultiVu.dll	Library used by MultiVu containing code for the moment measurement	
C:\QdSquidVsm\SVsm\System	DCScanProcess.dll	Library for performing data analysis (fitting) of DC scan data	
C:\QdSquidVsm\SVsm\System	seqtrans_vsm_....dll	Library for automatic updating of sequences written in older versions of MultiVu	
C:\QdSquidVsm\SVsm\System	Vsm.ini	INI file for customization of the measurement library	
C:\QdSquidVsm\System	*Service.exe	Windows Services that provide system control (e.g., temperature) even when MultiVu is not running	
C:\QdSquidVsm\System	*StateMachine.dll	Libraries used by the above services	
C:\QdSquidVsm\System	QdWapiLog.txt QdWapiLog_Bak.txt	Log files containing diagnostic information for the services/state machines	
C:\QdSquidVsm\Tools		Various utilities for system diagnostics	

## 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 interface allows immediate control of the MPMS 3 for performing a wide variety of standard tasks, such as changing the temperature or magnetic field, running measurements, or logging system data. The interface also allows most functions of the MPMS 3 and its measurement options to be automated by using a series of simple commands, called a sequence. Section 2.1.2 below describes how these commands are created and executed using a sequence editor.

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 3 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 MPMS 3 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 MPMS 3 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 MPMS 3 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

The MPMS 3 MultiVu menu options and toolbar buttons provide for multiple ways of performing many basic tasks, such as opening files and running sequences. The menu options and toolbar buttons that are available depend on the active file and the task being performed and also on the installed hardware options.

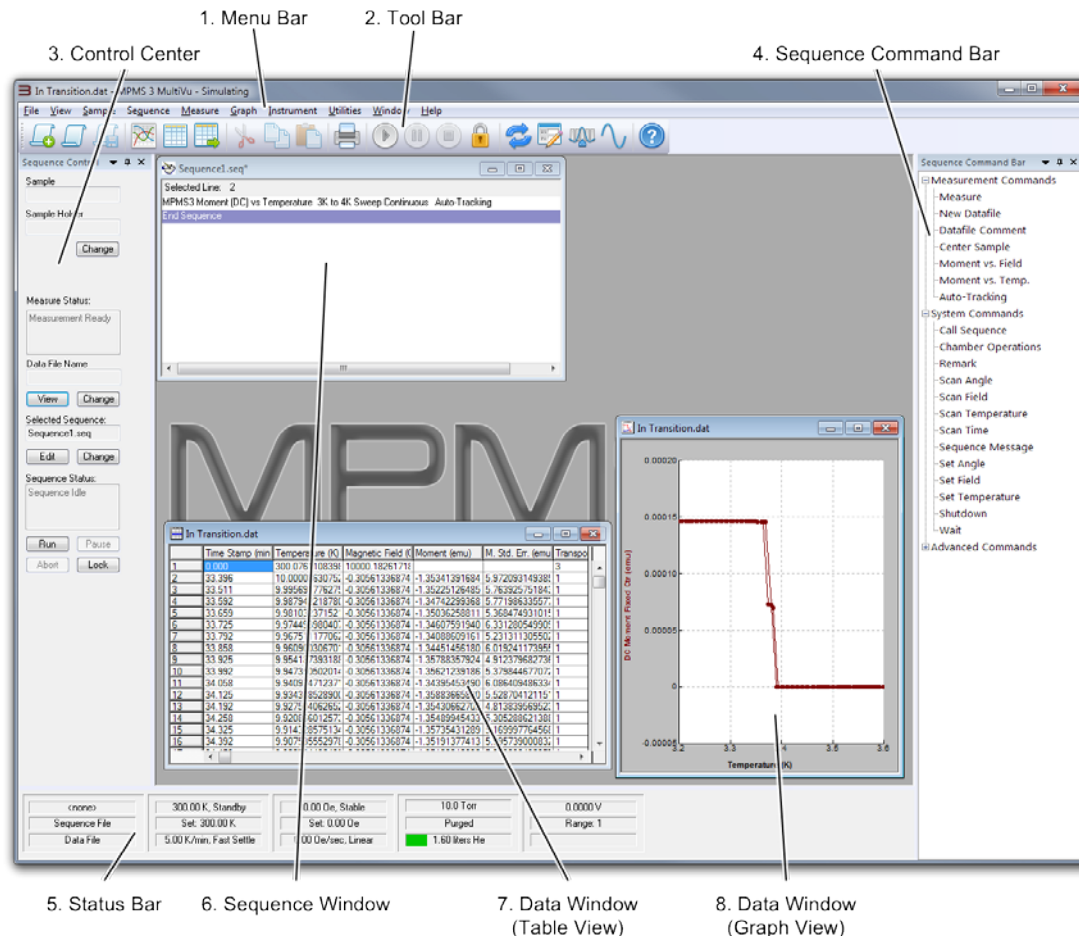


Figure 2-4. User interface overview.

The main MultiVu window is shown above. The tool bar, control center, sequence command bar, and status bar may each be docked and undocked or set to auto-hide by right-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. The available menus and menu options vary, based on the current task, active file, and installed MPMS 3 options. Some options add additional items to the menus.

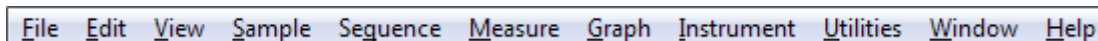


Figure 2-5. Main menu bar.

- **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 3, and access to the user documentation.

## 2.2.2 Tool Bar

The Tool Bar contains shortcuts that let the user initiate many of the more frequently performed MPMS 3 MultiVu functions without having to select multiple, nested menu options. The buttons are grouped according to function. The active file and the current state of the sequence operation determine which buttons are enabled.

The user may move, dock, hide, or display the tool bar. The “View > Tool Bar” toggle option hides or displays the tool bar.

If the mouse pointer pauses over a tool bar button, a ToolTip pops up display the name of the button.

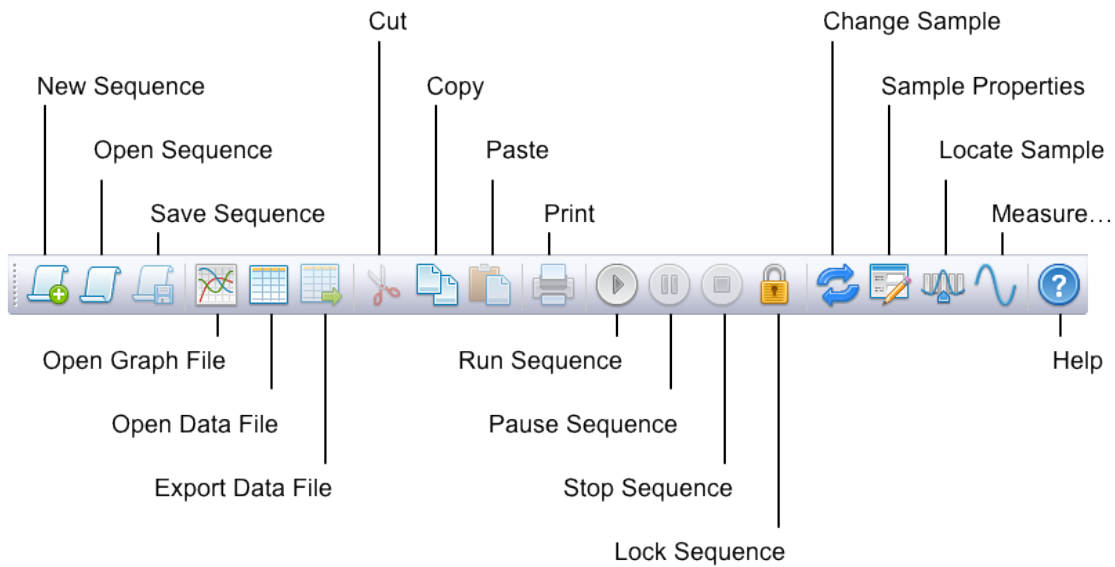


Figure 2-6. Tool bar.

### 2.2.3 Control Center

The Control Center (Fig. 2-7) 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.

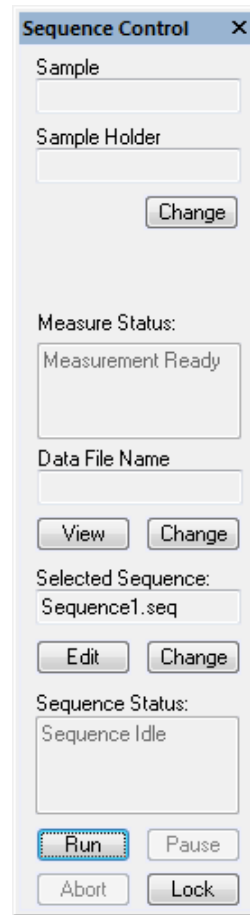


Figure 2-7. Control center.

### 2.2.4 Sequence Command Bar

The Sequence Command Bar (Fig. 2-8) 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 section 2.3.

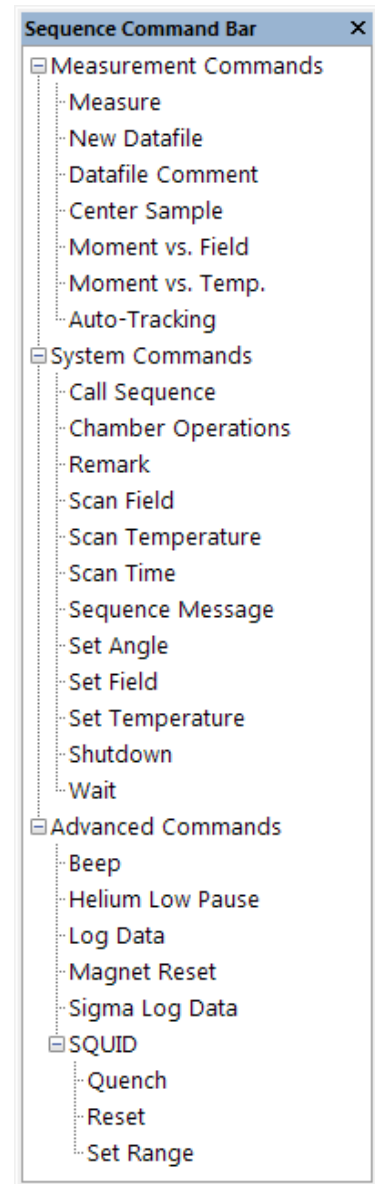


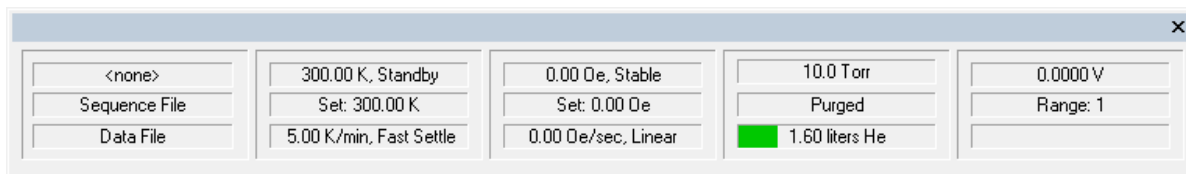
Figure 2-8. Sequence command bar.

### 2.2.5 Status Bar

The Status Bar displays the sequence status and the status of the magnetic field, system temperature, and the MPMS 3 sample chamber. 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.



You might minimize, maximize, hide, display or move the status bar. By default, the status bar is maximized to show the maximum amount of status information. The “View > Status Bar > None” menu entry hides the status bar. “View > Status Bar > Maximum” displays the entire status bar.



<u>Sequence:</u>	<u>Temperature:</u>	<u>Magnetic Field:</u>	<u>Sample Chamber:</u>	<u>SQUID:</u>
-status	-present state	-present state	-pressure	-reading
-file name	-setpoint	-setpoint	-state	-range
-execution line	-rate and mode	-rate and mode	<u>Helium Tank:</u>	
			-approximate volume remaining	

Figure 2-9. Status bar.

The sequence status panels indicate whether a sequence is running. The top or first panel always indicates the sequence status: Idle, Running (green background), or Paused (yellow background). During the run, the sequence status panels display the current sequence step, the name of the running sequence, and the base name of the active data file. If another sequence is selected for editing while a sequence is running, the sequence status panels continue to show the name of the running sequence and the base name of the data files storing the measurement data from the current sequence run. Clicking on any sequence status panel displays the control center.

The temperature status panels display the current system temperature and indicate the state of the temperature control. Clicking on any temperature status panel opens the Temperature Parameters dialog box. The field status panels display the current magnetic field and indicate the state of the field control. Clicking on any field status panel opens the Magnetic Field dialog box.

The chamber status panels display the state of the sample chamber status and indicate the level of helium in the dewar. Clicking on any chamber status panel opens the Chamber dialog box.

The SQUID status panels show the current SQUID voltage and range (1, 10, 100, or 1000) – during a SQUID reset or while tuning the SQUID the bottom panel will show SQUID status information. Clicking on any SQUID status panel opens the SQUID diagnostics window.

## 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. Hovering the mouse over any data point will display a tool tip with the values for that point. 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 section 2.4 “Graphing Data Files.”


---

## 2.3 Sequences

Operation of the MPMS 3 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” (Fig. 2-10). Enable a disabled command with “Edit > Enable.” 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.
- All of the commands found in the “Edit” menu are duplicated in a pop-up menu when you right-click any sequence window.

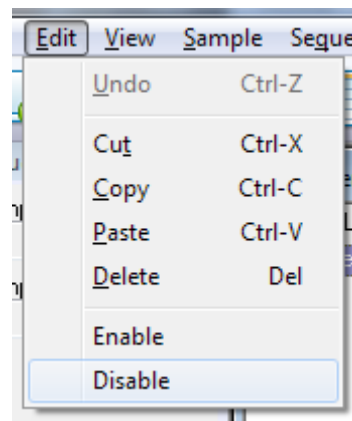



Figure 2-10. Disabling sequence commands.





### 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. If a sequence is aborted, it will finish its current measurement motion before executing the abort sequence command.

### 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.

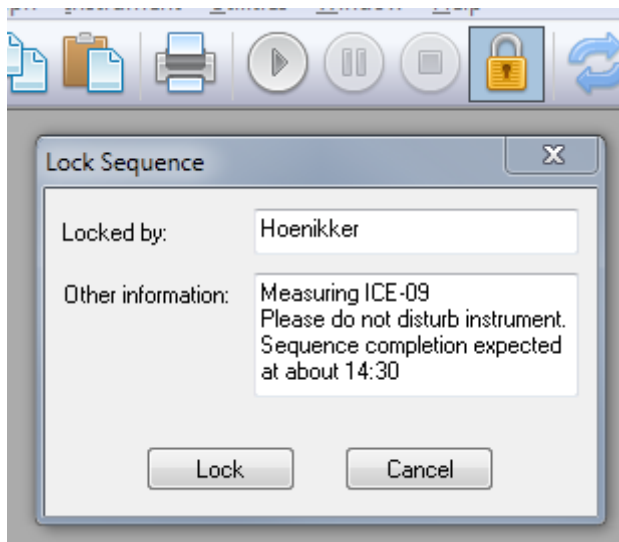



Figure 2-11. Locking/unlocking sequences.

- 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 3 may provide additional sequence commands not listed here.

### 2.3.5.1 MEASUREMENT COMMANDS

- **Measure** – Creates a sequence step specifying all necessary parameters (see tabs in Figure 2-12 below) to run or stop a measurement.

*Note that there might be additional tabs available between the “DC” and “Advanced” tabs that are not shown in the screen shots –tabs are added dynamically when additional measurement options (such as VSM or AC) are present. Please refer to the respective option manuals for details on the parameters available.*

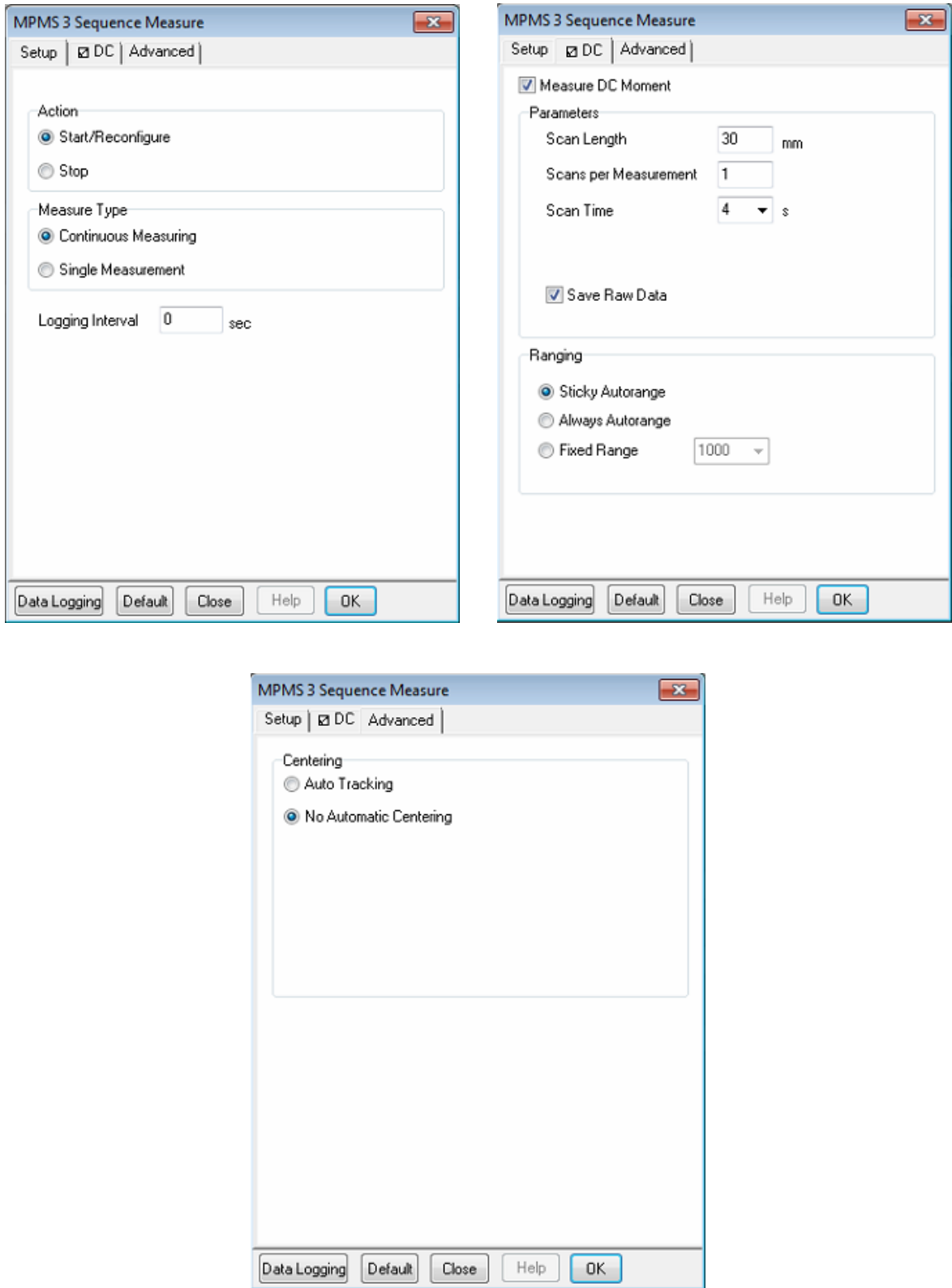


Figure 2-12. MPMS 3 Sequence Measure - measurement parameters interface.

- **Setup Tab**
  - **Action** – Select “Start/Reconfigure” to start measuring with the parameters specified on the other tab(s). Select “Stop” to stop any measurement that is currently running  
*Note: when “Stop” is selected as the action, none of the other settings in this or any other tab matter*
  - **Measure Type** – Specify “Continuous Measuring” for a continuous stream of data, or “Single Measurement” to collect a single data point  
*Note: when “Continuous Measuring” is selected, the sequence will immediately advance to the next step and the system will keep measuring until it encounters the next “Stop Measure” command. When “Single Measurement” is selected, the sequence will continue to its next command only after the data point has been acquired*
  - **Logging Interval** – Specify the logging interval (for continuous measurement stream) – specifying any number smaller than the measuring time for the selected measurement will result in every data point acquired to be logged to the data file as fast as possible.
  
- **DC Tab**
  - **Measure DC Moment** – Checkbox to en-/disable DC scan data acquisition. The status of this checkbox is also displayed in the title “DC” tab
  - DC scan parameters common to both Immediate and Automatic measurements (**Scan Length, Scans per Measurement, Scan Time, and Ranging**) are described in Section 1.4.4 Measuring Samples.
  
- **Advanced Tab**
  - **Centering** - Specify which automatic centering method is used to keep the sample centered (“Auto Tracking” typically gives the best results and is the preferred selection)
  
- **Data Logging** – Specify additional data that will be logged with each data point in the measurement data file
  
- **Default** – Reset all parameters to their system defaults
  
- **New Datafile** – Changes the data file being used to record data (Fig. 2-13). 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.

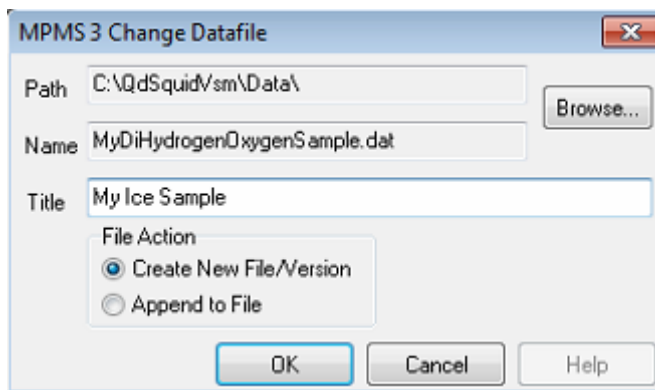


Figure 2-13. Creating a new datafile.

- **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.  
*This can be useful to tag data in order to facilitate post processing*
- **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.
- **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 in between fields and whether the field stabilizes for each measurement or continues changing (sweeps) while the measurement is performed.  
*Note that measuring while sweeping the field will result in decreased sensitivity and less accurate data and is only suggested for qualitative analysis. Moreover, due to coupling from the magnet to the superconducting gradiometer, the field sweep rate cannot be set too high or no data can be acquired as the SQUID will be over-ranging too often.*



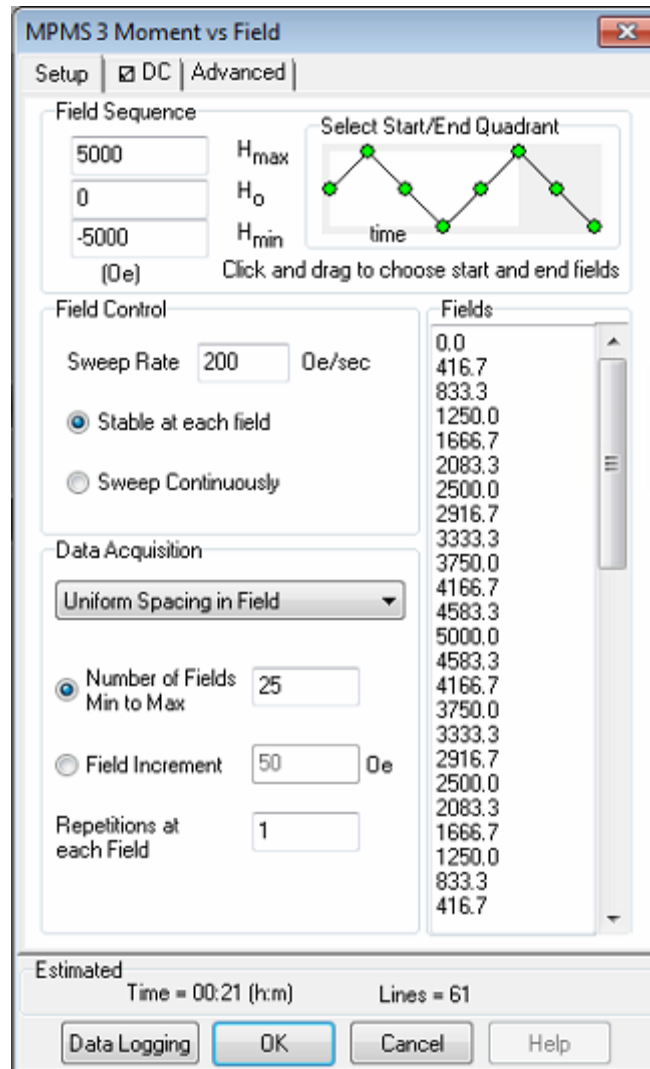


Figure 2-14. MPMS 3 Moment vs. Field sequence command - setup interface.

You specify 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 software displays the approximate magnetic fields where measurements will be performed with your settings, and the estimated total time to perform the measurement series.

Measurement parameters and selection of measurements to be performed can be specified in the individual measurement tabs in the same manner as for the “Measure” command above.

*Note that the “Moment vs. Field” command requires that the temperature is stable at each field before collecting any data.*

- **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 data spacing in temperature (T) (uniform in T, T<sup>2</sup>, T<sup>1/2</sup>, 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 software displays the approximate temperatures where measurements will be performed with your settings, and the estimated total time to perform the measurement series. Measurement parameters (e.g., scan length, scan time) can be specified in the individual measurement tabs in the same manner as for the “Measure” command above (see chapter 4 for application considerations). In addition to Auto-Tracking settings, the “Advanced” tab also allows you to specify 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.
- **Auto-Tracking** – Allows you to turn Auto-Tracking on or off. Note that Auto-Tracking should typically be enabled as it will provide the most accurate data by keeping the sample automatically centered while changing temperatures – Auto-Tracking might be disabled when using custom sample holders where no Auto-Tracking parameters are available.

### 2.3.5.2 SYSTEM COMMANDS:

- **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. 2-15)
- **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 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<sup>2</sup>, H<sup>1/2</sup>, 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, and remain stable, until the commands within the loop are completed. A small amount of field overshoot can occur in this mode.

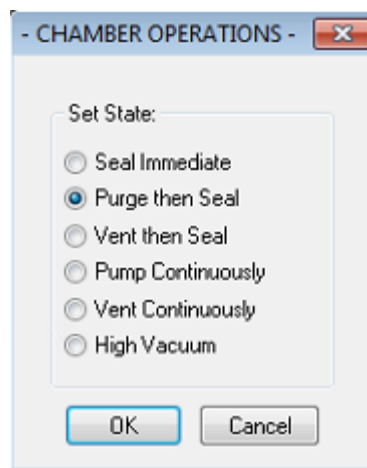


Figure 2-15. Chamber operations menu.

- *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, and remain stable, 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, and remain stable, 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.

*Note that measuring while sweeping the field will result in decreased sensitivity and less accurate data and is only suggested for qualitative analysis. Moreover, due to coupling from the magnet to the superconducting gradiometer, the field sweep rate cannot be set too high or no data can be acquired as the SQUID will be over-ranging too often.*

- **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, and remain stable, 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 set point, and will then slow the rate significantly to avoid temperature overshoot. At each set point the temperature will stabilize, and remain stable, 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 set point, 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 cooling flow is reduced while the current temperature is maintained.
- **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.)

### 2.3.5.3 ADVANCED COMMANDS:

- **Beep** – Causes the computer to make the Windows default beep sound.
- **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 – this emergency level is 0% in an EverCool system) (see section 1.6).
- **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.
- **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.)
- **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.
- **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.

## 2.4 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...”

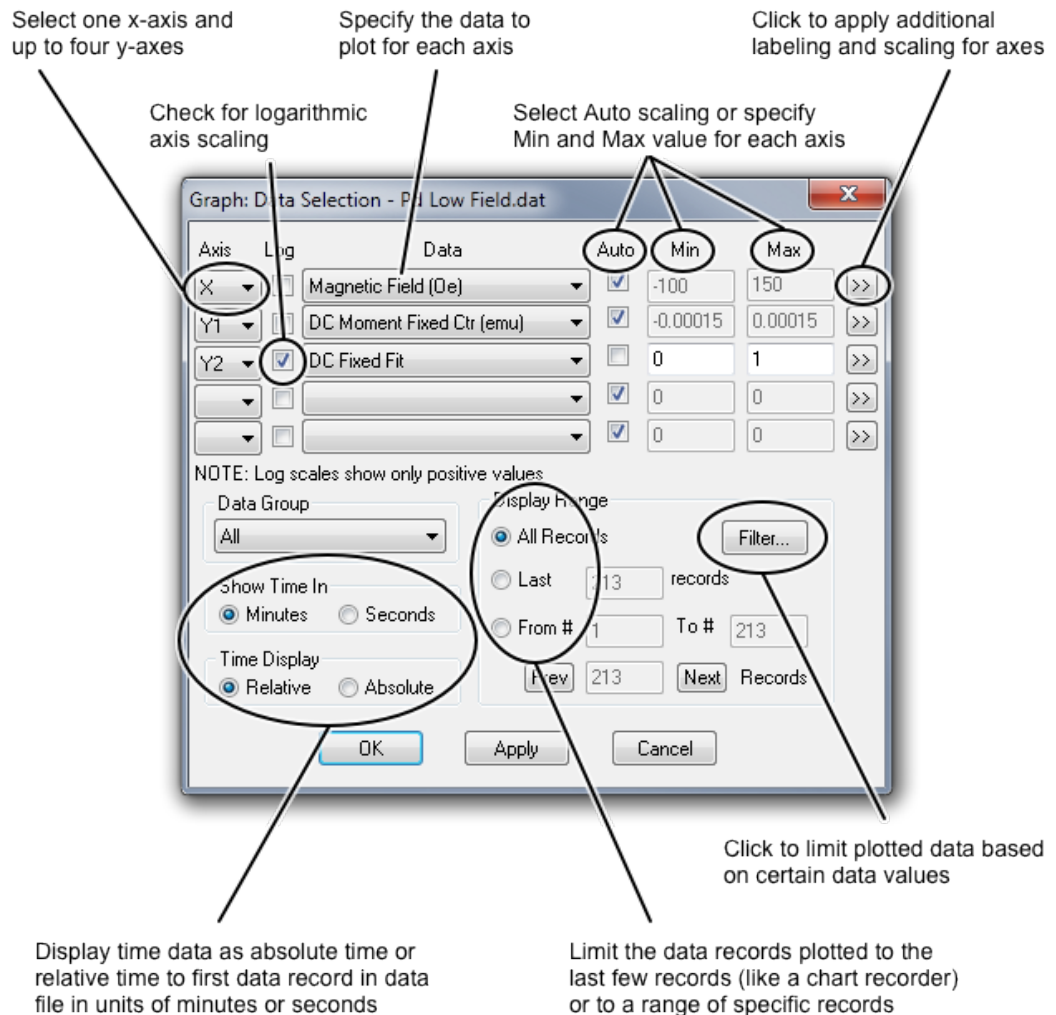


Figure 2-16. Graphing parameters interface.

- 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.
- To see more data in an additional window, right-click on the existing graph view window and select **>>New Window**.
- 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 fit quality to hide data with a bad fit (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.

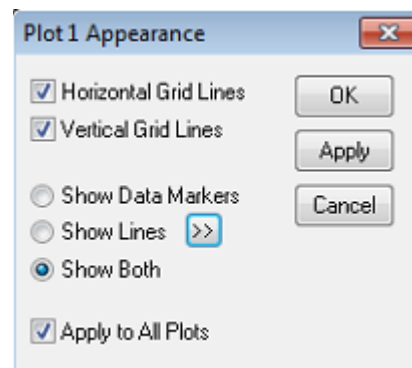


Figure 2-17. Plot appearance interface.

### 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 MPMS 3 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 the “Export Data...” toolbar button () or choose “File > Export...” from the menu. This feature generates data files from the data you specify in several formats that most graphing and spreadsheet programs understand.



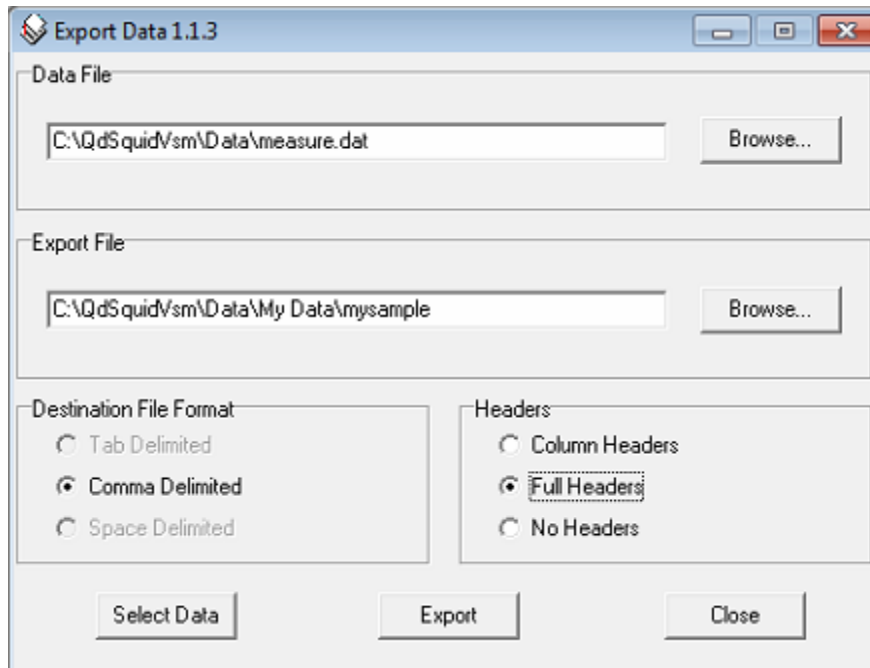


Figure 2-18. Exporting data.

- 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.

## Hardware

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### 3.1 Overview

**In this chapter** we describe the MPMS 3 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. 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) scanning 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.

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### 3.2 System Setup

The MPMS 3 hardware is primarily contained in a two-part metal enclosure. The upper half of the enclosure contains a computer running the MultiVu software and a module bay housing the instrument's modular electronics. The lower half of the cabinet contains a dewar. The sample transport is mounted on top of the cryogenic insert. The lower end of the insert contains the shielded SQUID, a 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.

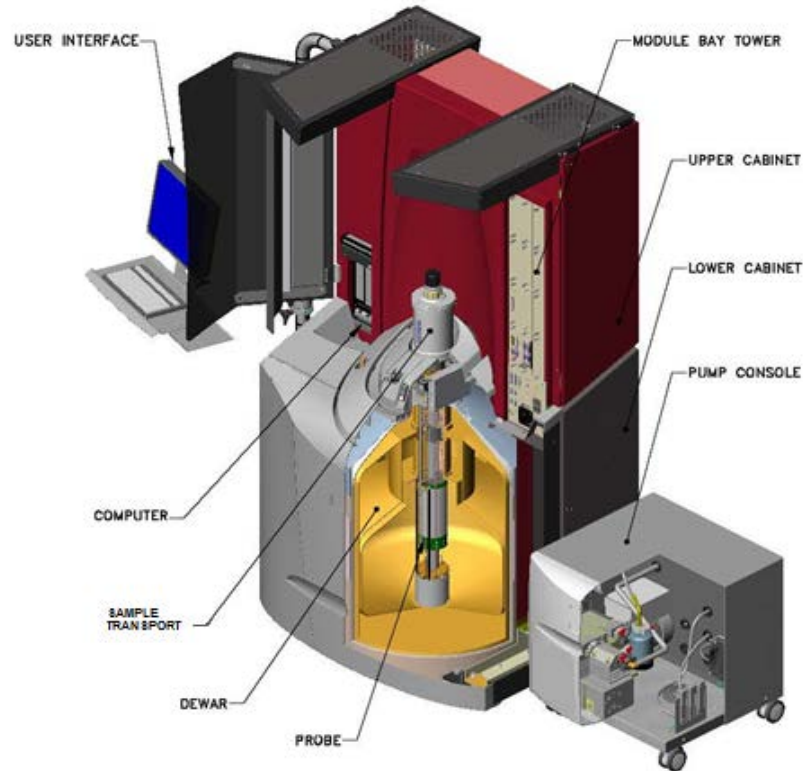


Figure 3-1. General system setup.

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 (35 in) 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 helium compressor is required which is powered by a separate grounded 3-phase power cord.) The MPMS3 requires 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 C.3.




#### **WARNING!**

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

Always power the system down before unplugging the power cord.

 **WARNING!**

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.

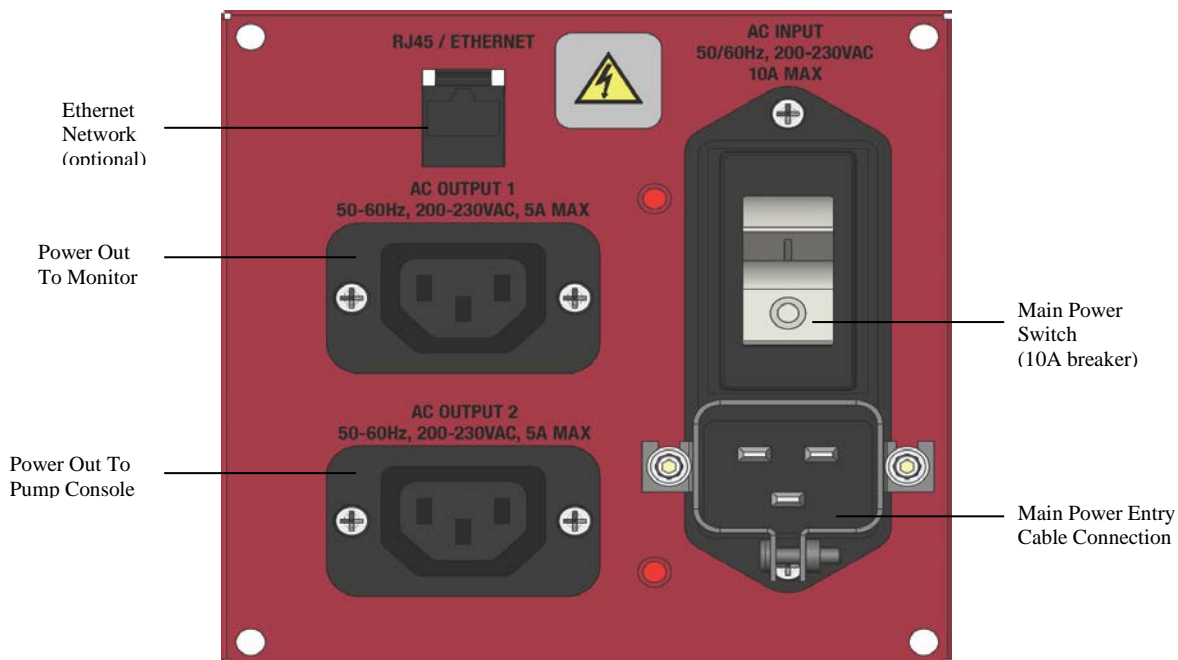


Figure 3-2. Power distribution unit.

In addition to the main MPMS 3 enclosure, a pump cabinet houses a vacuum pump required for instrument operation. 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 application software and low level application services 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)

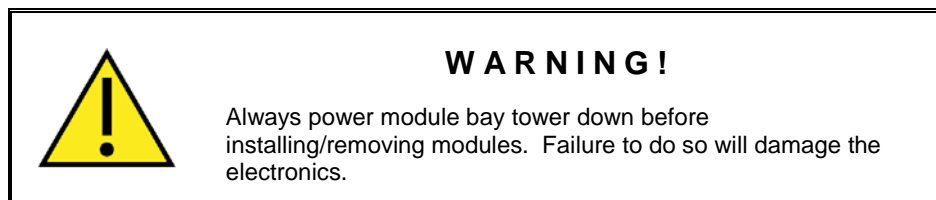
## 3.2.2 Modular Electronics

The electronic components of the MPMS 3 are housed in modular enclosures that are easily added to the system or replaced in case of a system 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) bus.

The basic MPMS 3 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 (Figure 3-3) that provide power and CAN bus interfaces in a standardized format. Additional controllers supplied with upgrade options may also be housed in these bays.



### 3.2.2.1 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 (Figure 3-4): low power modules have two jacks, high power modules have one jack.

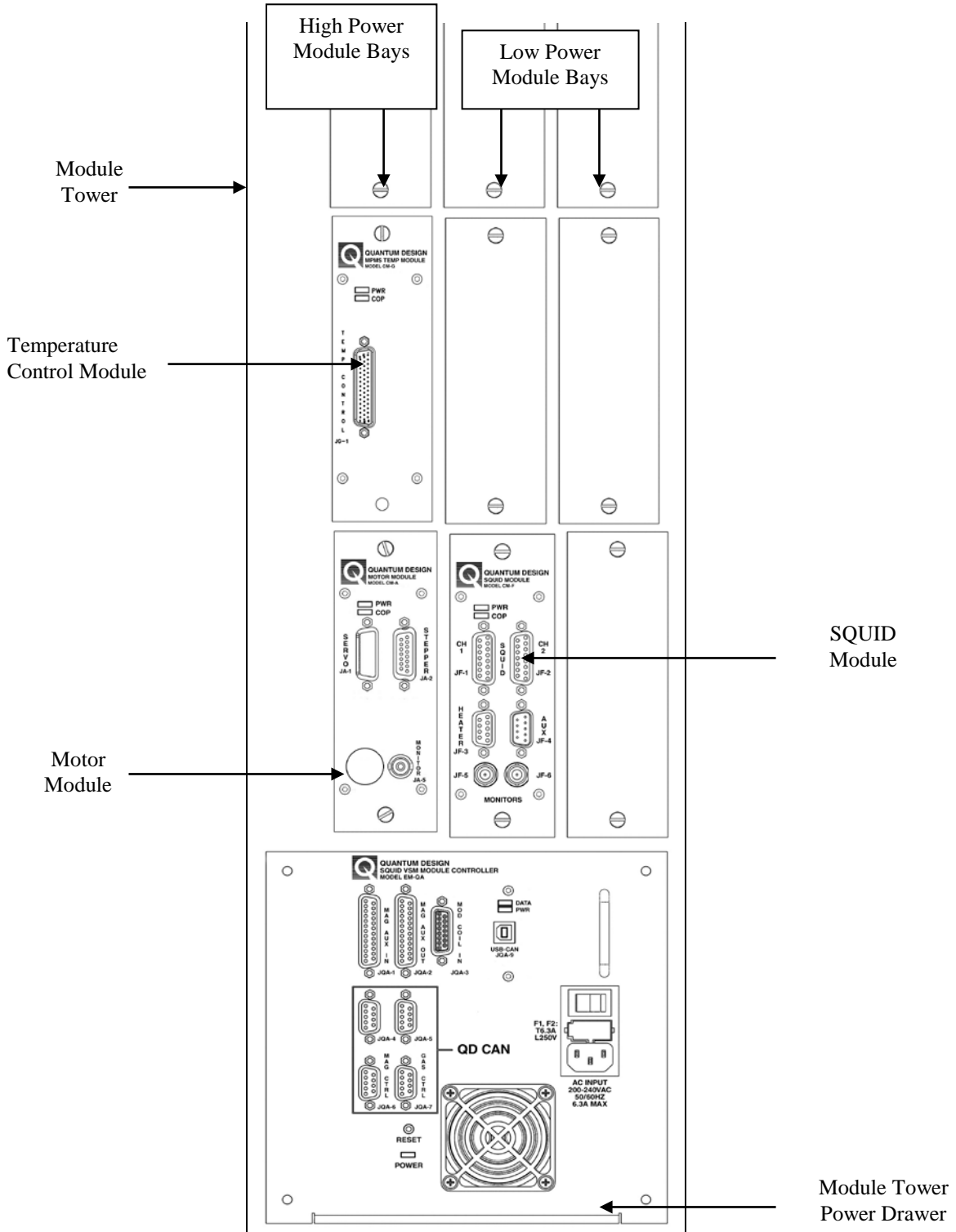


Figure 3-3. Module bay tower.

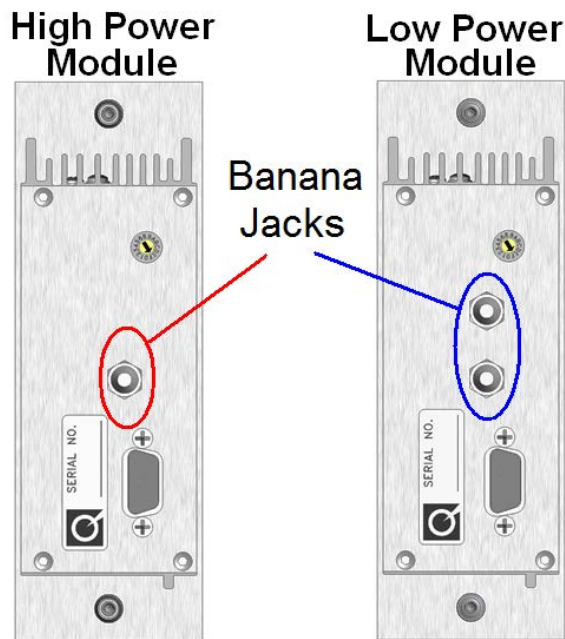


Figure 3-4. Module rear panels.

The panel below the module bays provides access to the power supply tray (Model EM-QA). 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 four unpaired fuses are behind the panel of the power supply tray and should only be replaced with equivalent 5A, 20 mm, delay fuses under the direction of a qualified Quantum Design service representative (Figure 3-5). 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.

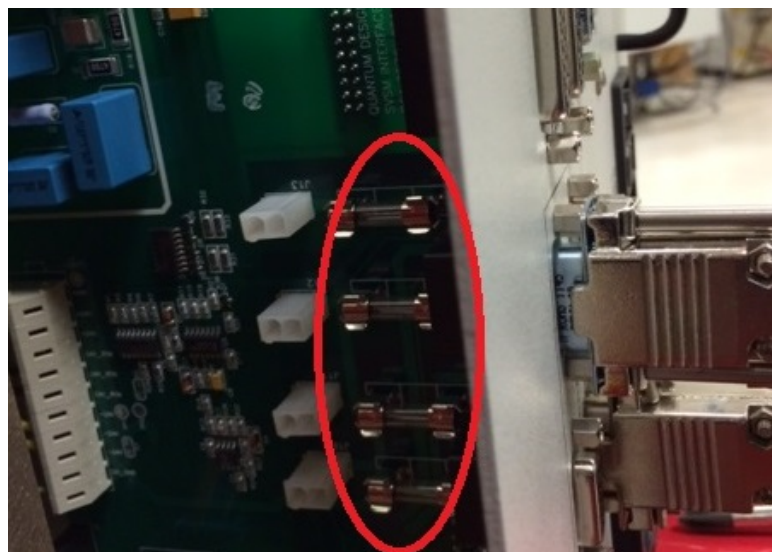


Figure 3-5. Location of the four unpaired fuses (5A, 20mm) for the CAN-based electronics



The model EM-QA module controller also utilizes two 6A, 20 mm delay fuses at the power entry (Figure 3-6). 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.



Figure 3-6. Location of the two, paired delay fuses (6A, 20mm) at the CAN tower power entry

### 3.2.3 Cryogenic Equipment

#### WARNING!



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 closed room. (4) Liquid or vapors may backstream out of fill ports when removing caps/plugs/transfer lines.

#### WARNING!



To guard against these dangers, wear protective clothing such as gloves, goggles, 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 3. Tanks, tubes, and hoses for these cryogenics 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 cryogenics. 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 3. 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. (Liquid nitrogen is not used for MPMS 3 EverCool systems - refer to the EverCool option manual). 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.

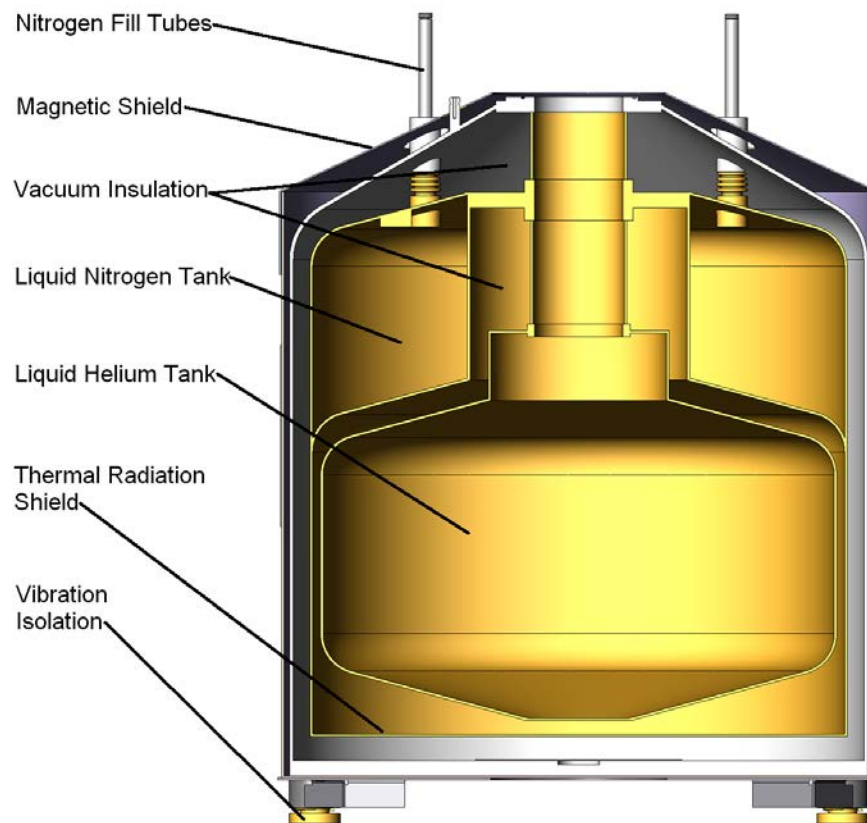



Figure 3-7. MPMS 3 dewar.

**WARNING!**



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 barb on the back of the main cabinet through 0.2 psi (1.4 kPa) relief valves. Rubber hoses 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. Make sure to terminate ground loops that may be caused when connecting a recovery system to the MPMS 3.

The N2 exhaust rubber hose features an inline one-way valve 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 performing a HELIUM transfer. If this is the case, please contact QUANTUM DESIGN. There might be risk for plugging the N2 exhaust with ice.

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## 3.3 Temperature Control System

Temperature control for the MPMS 3 is achieved with several electronic and software components, allowing stable and continuous operation from 1.8 to 400 K with the standard system, including smooth temperature control through the 4.2 K liquid helium boiling point. Temperature control is orchestrated by the Temperature Service on the computer. The Temperature Service software receives the temperature set points and then sends commands to the temperature control electronics to achieve the desired result.

Two electronic modules are involved in temperature control:

- Temperature Control Module (model CM-G, located in the CAN module bay)
- Gas Handling Controller (model EM-QD, integrated into the top of the probe).

This section presents brief details of the components and operation of the temperature control system.

## 3.3.1 Temperature Control Electronics and Software

### 3.3.1.1 TEMPERATURE CONTROL MODULE (TCM)

The MPMS 3 sample chamber has a number of heaters and thermometers attached to the outside. Three Negative Temperature Coefficient (NTC) thermometers are used for temperature sensing. The primary sensor (sample thermometer) is located at the null point of the SQUID pickup coil. Two other sensors, gas and neck thermometers, are located at the bottom of the sample tube and between the sample thermometer and the top of the sample tube. Corresponding heaters are located near each thermometer. Because of the importance of the sample temperature accuracy, the temperature of the sample thermometer is calibrated in-situ in the system. Since strong magnetic fields can influence the accuracy of the thermometer at temperatures below 15 K, the sample thermometer is also calibrated under magnetic fields. The temperatures of the gas and neck thermometers are pre-calibrated prior to installation on the sample tube. The temperature control module reads out the gas thermometer, sample thermometer, and neck thermometer on the probe. The TCM also provides heater current to the gas heater, sample heater, and neck heater based on feedback temperature control using thermometer readings.

Temperature is regulated by PID (Proportional-Integral-Differential) control. The PID control is optimized depending on the temperature range of interest (Table 3-1). Between 320 K and 400 K, PIDs for the sample and gas thermometers are active. Going down in temperature from 320 K to near 10 K, the PIDs for the sample and gas thermometers are active. Going down from 10 K to 1.8 K and going up from 1.8 K to near 15 K, only the PIDs for the sample thermometer are active. Going up from 15 K to near 320 K, the PIDs for the sample and gas thermometers are used, while the PIDs for the neck thermometer is active between 100 K and 320 K.

Three thermometers are assigned to three of four bridges in the module, where the fourth one is a spare; SIMM interface 0: sample, SIMM interface 1: gas, SIMM interface 2: neck, and SIMM interface 3: spare (no simm card installed).

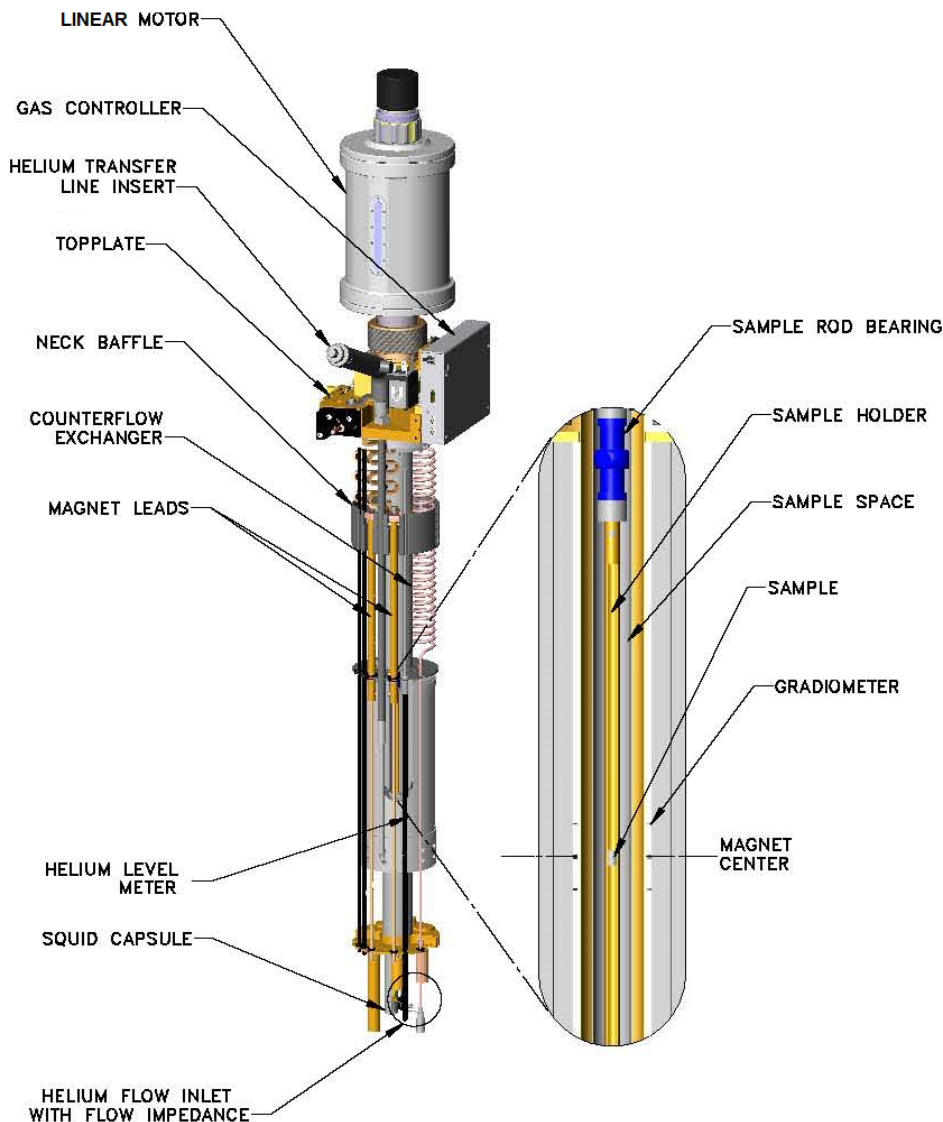


Figure 3-8. MPMS 3 probe.

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.

### 3.3.1.2 GAS HANDLING CONTROLLER (GHM)

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 (GHM) electronics control the mass flow controller for the main cooling flow and the impedance heater. The GHM electronics also turn on and off the various solenoid valves as needed and – for systems equipped with the oven option – interfaces with the HiVac pump. Additionally, it monitors the helium level within the cryostat and reads out the sample pressure from the pressure sensor located at the top of the probe. The GHM

Utility (Figure 3-9) is the user interface for monitoring such things as CFE flow, heaters, and helium level, and executing chamber operations and valve states. For illustrative purposes, heaters and thermometers have been drawn on the GHM Utility window to show their approximate locations. The chamber heater and thermometer locations align with the position of a sample placed in the chamber, at  $66\pm 3\text{mm}$ .

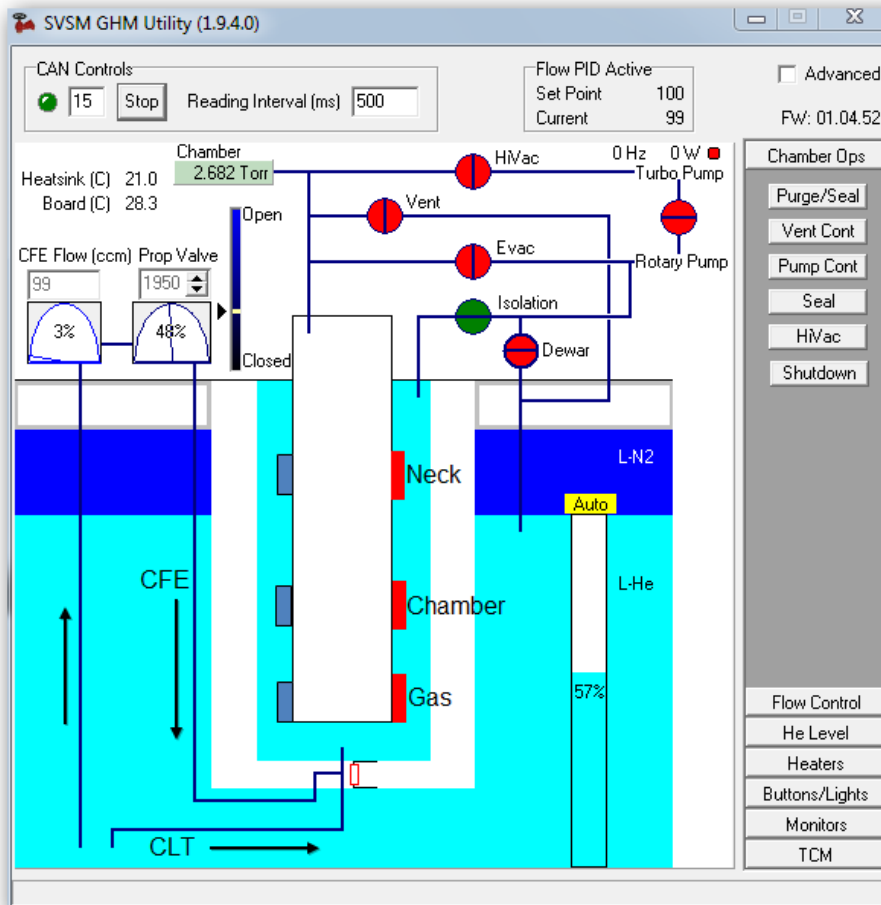


Figure 3-9. Gas monitor interface.

### 3.3.1.3 COOLING POWER

Cooling power is provided by allowing a controlled amount of liquid helium from the dewar to enter the bottom of the cooling annulus. The liquid helium enters through a variable impedance inlet (counterflow exchanger; CFE) and a fixed impedance (continuous low-temperature (CLT) inlet). The liquid helium turns into a gas, which flows through the cooling annulus and is pumped from the top of the probe by the vacuum pump. The CFE is controlled by a mass flow controller. During the cooling mode, the chamber heater is turned off.

At or near the temperature set point, longitudinal copper wires along the length of the sample chamber maintain thermal uniformity and several torr pressure of helium gas in the sample chamber provides thermal contact with the sample.

### 3.3.1.4 HEATING POWER

Temperature control within the sample space is achieved by heating and cooling the outside wall of the sample chamber. Exchange gas in the sample space transfers the wall temperature to the sample. The three heaters and temperature sensors (neck, sample, and gas) reduce temperature gradients within the sample space and allow for more accurate modeling of sample position versus temperature.

### 3.3.1.5 GAS FLOW

Cooling power is maintained by a continuous flow of helium from the bottom of the dewar, which is regulated by the external pump. There are two impedance tubes which regulate cooling: the counterflow exchanger (CFE) and the continuous low-temperature (CLT) capillary. The CFE flow operates in a range between 0.1 LPM and 5 LPM via a mass flow controller, enabling rapid cool-down to 10 K. The maximum cooling rate is 30 K/min, with typical cooldown from 300 K to 10 K (stable) in 15 minutes. Below 10 K CFE is closed completely and the CLT flow controls the cooling rate. The typical cooldown from 10 K to 1.8 K (stable) is within 5 minutes at cooling rate of 10 K/min.

To hold temperatures below 10 K, 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, where a scroll pump is used.) pumping through a solenoid valve that is used for isolating the region under special circumstances, such as power loss.

Occasionally, the CLT impedance 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 150 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 O-ring 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.

### 3.3.1.6 TEMPERATURE CONTROL STATES

In the MPMS3 Temperature Control System, the MultiVu display indicates whether the temperature is considered to be stable, near, or chasing (or tracking) to the set point. Table 3-1 shows how the temperature is controlled during the different modes of temperature state.

Table 3-1. Temperature Control System States

Cooling Mode										
	Thermometer PID			Heater Used			Helium Inlet		Gas Flow (cc/min)	
	Gas	Sample	Neck	Gas	Sample	Neck	CFE	CLT	CFE	CLT
400 to 320	Active	Active	OFF	Active	Active	OFF	Open	Open	100 to 5,000	250
320 to 10	Active	Active	Active	Active	Active	Active	Open	Open	100 to 5,000	250
10 to 1.8	OFF	Active	OFF	OFF	Active	OFF	Close	Open	0	250

Heating Mode										
	Thermometer PID			Heater Used			Helium Inlet		Gas Flow (cc/min)	
	Gas	Sample	Neck	Gas	Sample	Neck	CFE	CLT	CFE	CLT
1.8 to 15	OFF	Active	OFF	OFF	Active	OFF	Close	Open	0	250
15 to 320	Active	Active	Active	Active	Active	Active	Open	Open	100	250
320 to 400	Active	Active	OFF	Active	Active	OFF	Open	Open	100	250

If the temperature control system appears to be malfunctioning, and fails to reach or maintain the requested temperatures, it is useful to monitor various parameters. The software has capabilities to log diagnostic data to help identifying the cause of faults. If problems with the system recur frequently, you are encouraged to contact your Quantum Design service representative for assistance.

Note: When contacting your Quantum Design service representative for assistance, it is vital that you be as detailed as possible. Most problems cannot be solved without specific information. Please be prepared to provide as much of the following information as possible:

- Description of failure, temperature and frequency of occurrence
- Recent changes to the system, including helium fills, addition of new equipment such as helium recovery system, etc.
- Provide relevant information such as a sequence, data file, diagnostic log file, and Event log file, etc.

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## 3.4 Magnetic Field Control System

The magnetic field in the MPMS 3 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 QuickSwitch. The QuickSwitch 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 QuickSwitch 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. In addition, the QuickSwitch acts as a filter of external fluctuations from the power supply.

The magnetic field reported by the instrument is computed from 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*.



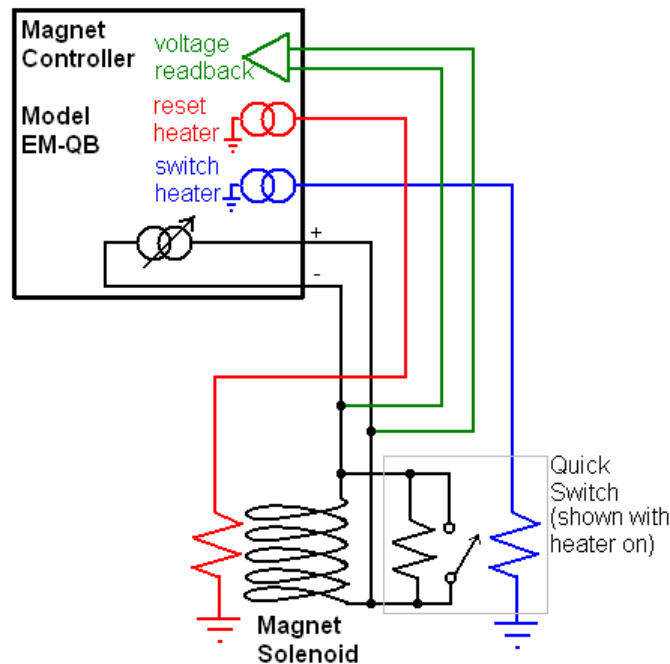


Figure 3-10. Magnet control diagram.

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 the system's utility kit.

### 3.4.1 Magnet Safety

No cables should ever be disconnected without first driving the magnet to zero field. The MPMS 3 superconducting magnet can trap magnetic flux and therefore can lead to a hazardous situation if a charged magnet is left completely unconnected to the system, as doing so leaves no way to discharge the magnet directly. Several different cables contain connections for magnet control. Be sure to drive the magnet to zero field before disconnecting any cables if the probe ever needs to be disconnected from the controllers.



### WARNING!

Never disconnect a charged magnet from the controllers. If the probe must be disconnected from the controllers, be sure to drive the magnet to zero field before disconnecting the controllers from the probe head.

High magnetic fields can be dangerous. Fields above 5 Gauss are potentially harmful to persons with medical implants (e.g. pacemakers, neurostimulators, etc.). The MPMS 3 is configured with an integrated Environmental Magnetic Shield, and the 5 Gauss line of the longitudinal magnet is fully contained within the system's enclosure. Therefore, with the MPMS 3 system, there are no safety concerns regarding stray fields.

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## 3.5 Motion Control System

The motion control system moves the sample within the detection coils. The sample transport, or motor, is a long-throw linear motor mounted on springs within its casing for vibration isolation (Figure 3-9). The sample transport receives current from the motor control module, model CM-A. A DC signal controls the sample position. A precision optical encoder in the sample transport 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. The armature position may be seen through a window on the front of the sample transport.

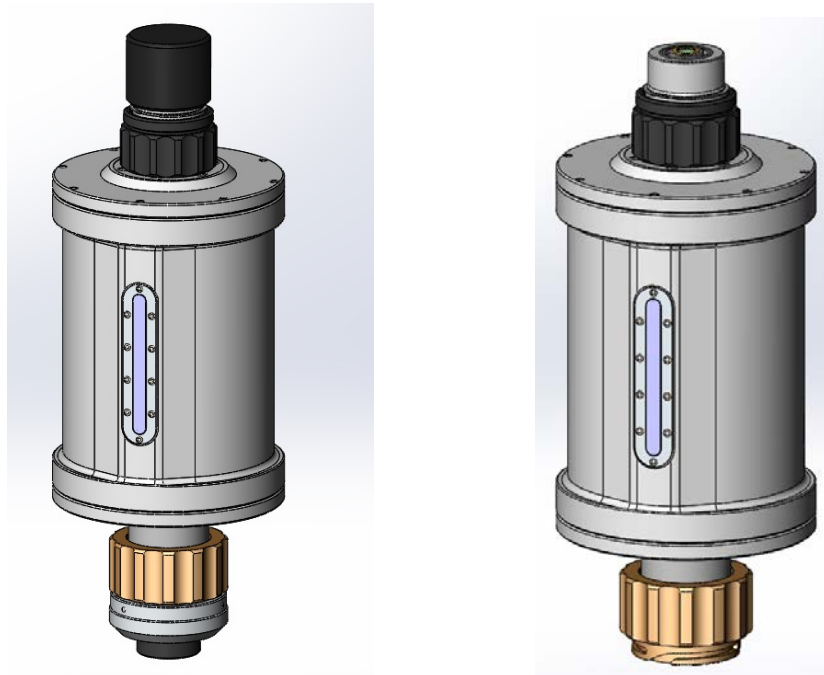



Figure 3-11. Sample transport with and without end caps.

In DC measurement mode, the sample is scanned up and down at constant speed, according to the measurement parameters set by the user. Refer to Table 1-1 for a summary of the DC measurement parameters

- The linear motor in the sample transport 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.
- For samples that cannot be exposed to the stray magnetic field of the sample transport a sample shield is available.

## 3.6 SQUID Detection System

**WARNING!**



**CAUTION:** SQUID detection is extremely sensitive to the magnetic signal generated by a user sample, but also to external electromagnetic disturbances, such as cell-phones, RF-furnaces or stray-fields of equipment in close proximity. Environmental noise could affect the performances of your system. We advise 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.

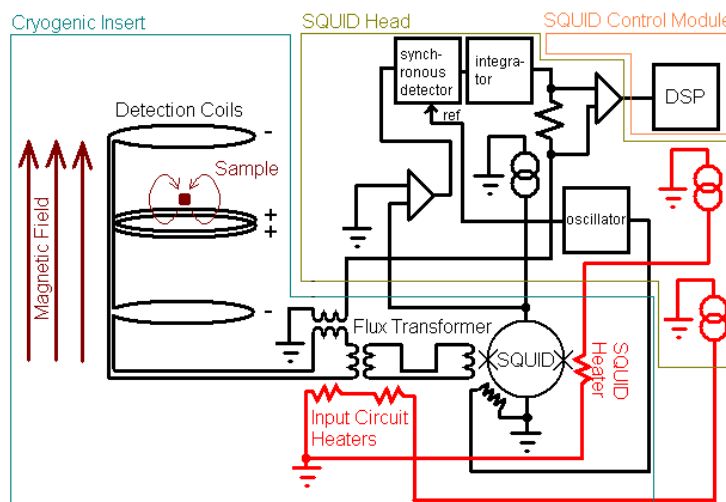


Figure 3-12. SQUID detection diagram.

Figure 3-12 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 moves the sample through the detection coil set (over the specified scan length), and locates where the SQUID signal peaks. (See Chapter 4 for more details on measurement theory).

- With this type of measurement it is extremely important that the sample offset be properly determined.

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 ( $\Phi_0$ ) 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. 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.

Over-ranging is generally not encountered during DC scans, with the exception of high moment samples. The DC scan is capable of measuring samples with moments as large as 5 emu. However, if the SQUID signal (in any range) exceeds 5 V, modifications to the measurement parameters or the sample itself may be necessary to mitigate over-ranging of the SQUID. The following will help to overcome the dynamic range limitations:

- Enable auto-ranging (if disabled)
- Measure at smaller field (if the signal is field dependent)
- Use a longer measurement time
- Measure a smaller amount of the sample, if possible

More detailed information regarding this issue can be found on the Quantum Design website, in *Service Note 1500-229: DC Scan and High Moment Samples*.

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## 3.7 Chamber Atmosphere Control System

During measurements, the sample chamber and sample transport 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 moving, 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 sample transport 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 sample transport 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 sample transport is open for removing or inserting samples, clean helium gas is allowed to flow through the sample transport 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 Sample transport 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.

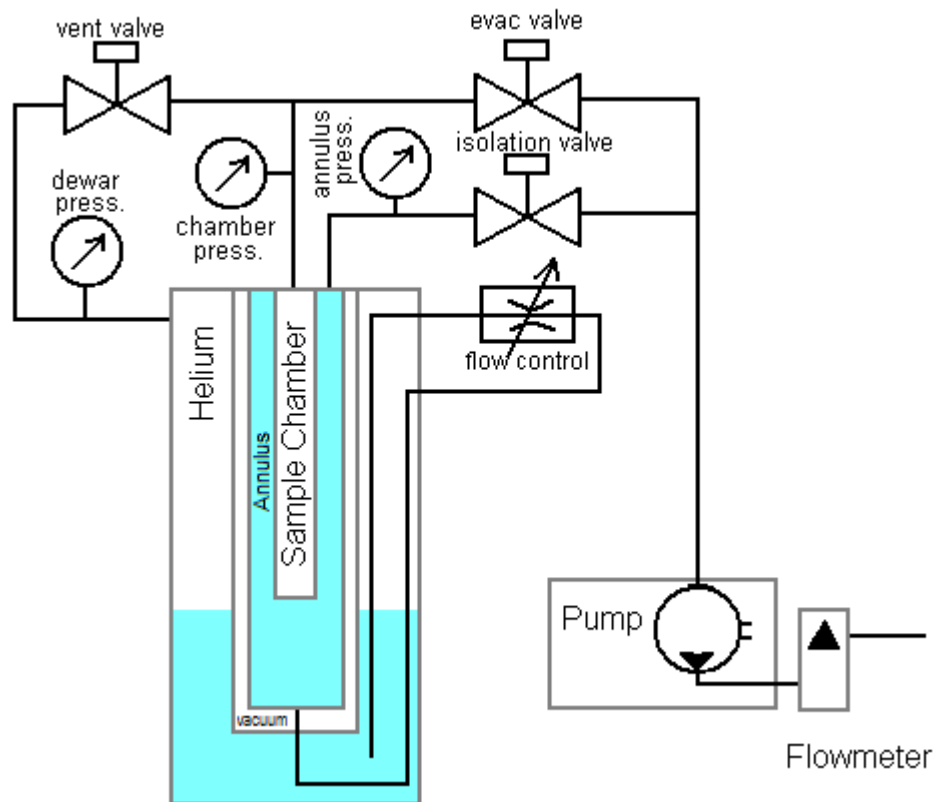


Figure 3-13. Gas handling diagram.

## 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. Dewar volumes and times are given for a standard MPMS 3, not an EverCool MPMS 3. In case of an EverCool system, refer to the EverCool option Manual.
- 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 3 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.

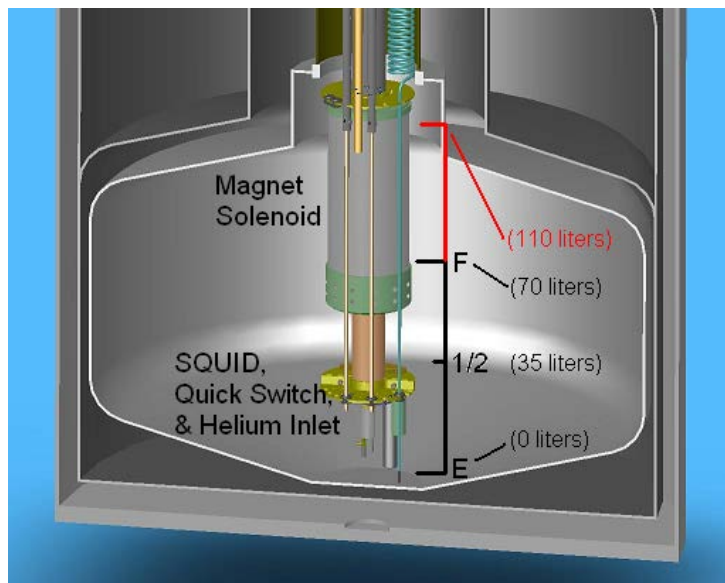


Figure 3-14. Helium level inside dewar.



### WARNING!

**CAUTION:** Carefully read Chapter 1, Section 1.5 for detailed instructions on filling nitrogen and helium.

## 3.9 Standby Mode

The standby function in the MPMS3 is designed to put the machine in a safe state between measurements. It has lower helium consumption than normal output, but still tries to ensure that there is adequate circulation to prevent the system from plugging. The standby mode can be accessed through both a menu command and sequence command as shown in the figures. Issuing the menu command will cause the system to immediately begin the process to enter standby. Using the sequence command will cause the system to go into standby at that point in the sequence. The procedure to put the system into standby mode in both instances is the same.

The operations performed when a system is going into standby mode are the following:

- Current measurements are stopped and the motor is returned to the bottom position
- Magnetic fields (greater than 100 Oe) are ramped down by the power supply
- If the AC or ULF options are present, fields in the modulation and trim coils in the magnet are turned off
- Maintains the current temperature if the temperature is between 100K and 315K.
  - If the temperature is below 100K, set 180K
  - If the temperature is above 315K, set 305K
- Reduce the flow through the primary impedance from 100ccm to 50ccm

Setting a new temperature will cause the system to come out of standby mode and be ready for the next measurement.

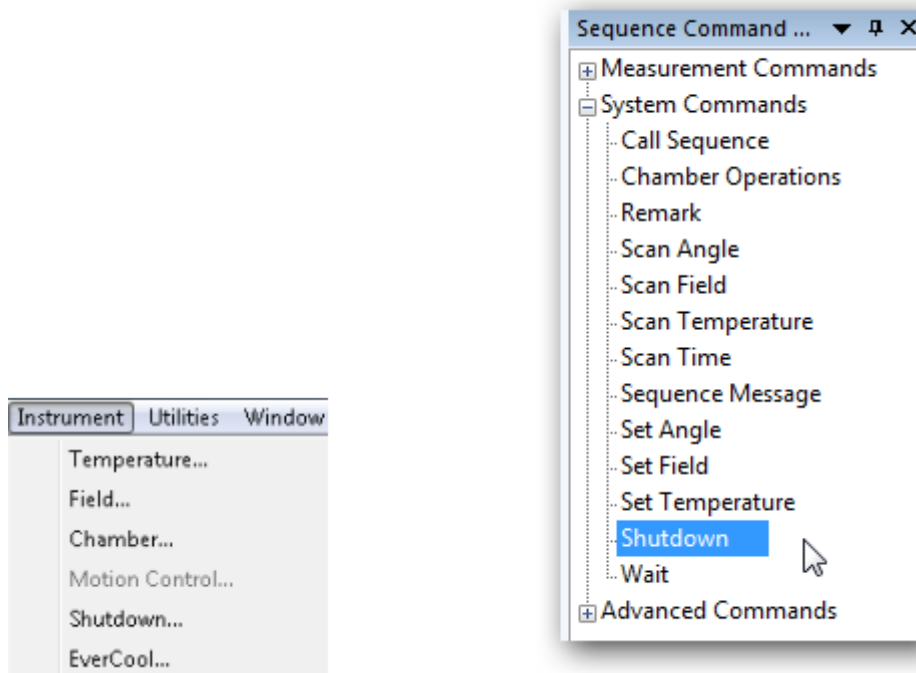


Figure 3-15. Initiating Standby Mode via the "Instrument" menu and by issuing a sequence command.

## 3.10 Additional Hardware

### 3.10.1 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 MPMS 3. 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 Sample transport. 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 acetone). 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 F for ordering replacement parts.)
- Inspect the flexible coupling (flexture) which is just beneath the magnetic latch. If damaged this will cause rattling and will produce noise in sample measurements. A more detailed view can be found in Figure 1-2.
- Inspect the bearing at the bottom of the sample rod periodically, 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)

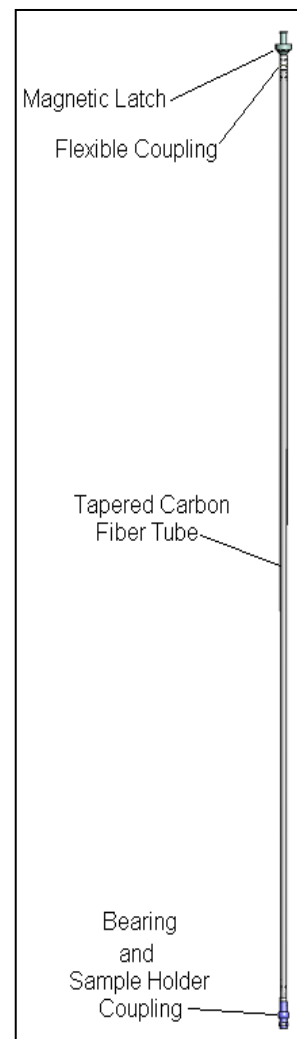


Figure 3-16. Sample rod.



### 3.10.2 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:

$$\mu = \chi_m H m,$$

where  $\mu$  is the magnetic moment of the sample,  $\chi_m$  is the mass susceptibility of palladium at 298 K ( $5.25 \times 10^{-6}$  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. A set of quartz sample braces. A pair of the quartz braces can be used to help 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. Two powder sample holders are used to safely encapsulate powder samples and snap them into brass holders. Application note 1096-306 discusses use of the powder sample 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.

# Measurement Theory and Applications

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## 4.1 Overview

In this chapter we give detailed insight into the measurement theory and the most common applications.

The MPMS 3 is a versatile and complex instrument which uses independent measurement techniques based on different data analysis methods to be seamlessly performed on a sample in the same environmental conditions with the same instrument. Optimizing the use of the instrument and obtaining the highest quality of data sometimes requires deeper knowledge that goes beyond the fundamentals of sample insertion and simple software operation. The sections below cover the theory of DC scan measurements (4.2) & squid drifts from various sources (section 4.3). In order to have the system perform at its highest performance, section 4.4-4.7 will cover the importance of sample geometry, sample holder effects, sample insertion & sample location effects. Section 4.8 will explain how to write an effective sequence using  $M(H)$ ,  $M(T)$ ,  $M(t)$ . Most common sources of errors will be found in section 4.9 and followed by further readings on Squid references in section 4.10

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## 4.2 Measurement Techniques

The MPMS 3 uses a DC scan method to measure magnetic flux in a superconducting loop as a function of sample position and fit using an analytic function. In addition to the DC scan, the optional MPMS 3-VSM option vibrates the sample sinusoidally and utilizes a lock-in amplifier to measure the magnetic response. The combination of these optional techniques allows for the optimization of the measurement method most appropriate for the experiment. The sample is ideally located in the center of the pickup coils, inductively coupling to the magnetic field and generating a current as it moves through the coil. The coil geometry is chosen to maximize the response of a sample oscillating inside the coil set Figure 4-1 shows a schematic of the cryogenic insert and the relevant position of the detection circuit.

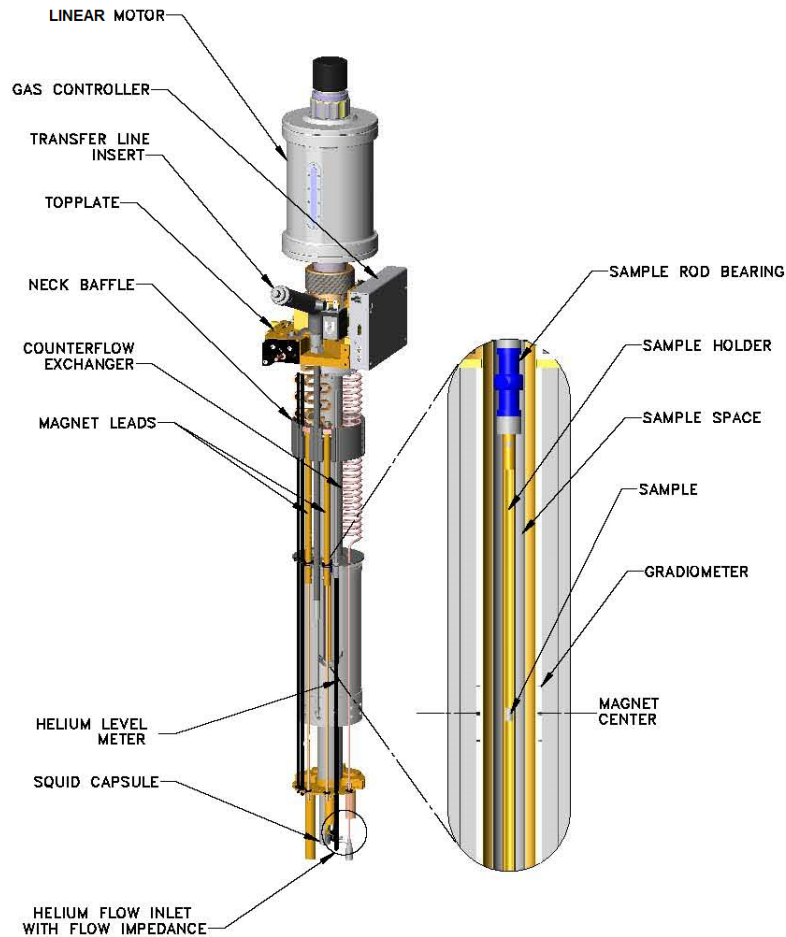


Figure 4-1. Cryogenic insert model.

The superconducting pickup coils are inductively coupled through an input transformer to a Direct Current (DC) Superconducting Quantum Interference Device (SQUID). The SQUID is located in a complete superconducting shield to prevent magnetic flux from the magnet or environment from creating noise. The advantage of the DC SQUID over a radiofrequency (RF) SQUID is that it enables much greater dynamic range from the detection circuit and negates the need for RF signals. Changing magnetic fields induce trapped flux inside the pickup loop that can lead to increased noise in the SQUID. Heaters are installed on the SQUID and the input transformer to remove trapped flux that can distort the measurement. In practice, this can be achieved by just quenching the input transformer rather than the entire SQUID chip. The input transformer, due to its construction, can be heated past its critical temperature in approximately 10ms and return to the superconducting state in a similar time frame. This feature enables very sensitive SQUID data collection within milliseconds of stabilizing at a magnetic field, producing fast and accurate magnetic field loop measurements. The applied field produced by the superconducting magnet is controlled by a hybrid power supply, which combines both analog and digital feedback to achieve field changes of 0.30e over the full field range. The accuracy of the applied field is limited by the trapped flux in the magnet and can be well characterized for each setup.

Figure 4.2 is a simplified schematic of the MPMS 3 detection hardware. The superconducting detection coils are configured as a second-order gradiometer, with counter-wound outer loops which make to first order the set of coils non-responsive to uniform magnetic fields and linear magnetic field gradients. Therefore a current is induced in the detection coils only in response to magnetic field disturbances.

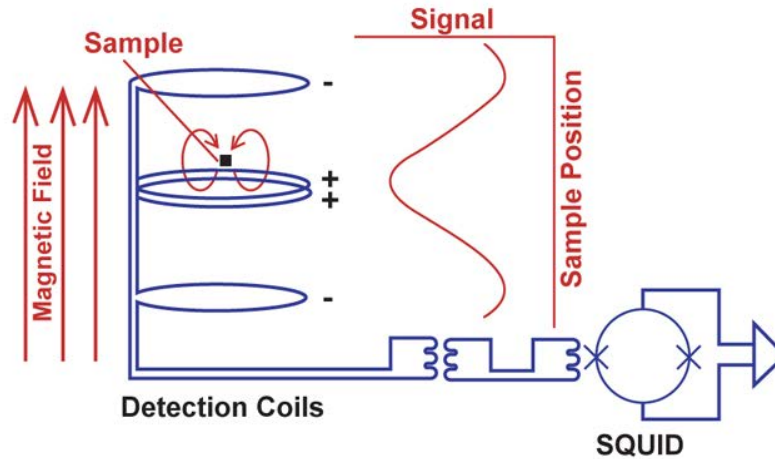


Figure 4-2. SQUID detection schematic.

Assuming the sample dimensions are much smaller than the dimensions of the detection coils, the current induced in the detection coils is a function of the sample position. The approximate shape of this function is shown above. In practice, 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. During a measurement the SQUID voltage is amplified and digitized by the instrument electronics.

### 4.2.1. DC Scan Measurement Theory

The DC scan fits the measured SQUID response as function of position to an expected response function. The top of Figure 4-3 shows a typical DC response function for standard sample geometry like the supplied palladium reference sample. For the DC scan, we collect two scans per waveform, both in the up and down motion. The motion path that we have chosen for the DC scan mode is based on a triangle wave. In contrast to sinusoidal motion that has a maximum velocity around the center of the coils where most of the information is gathered about the sample, the triangle wave gives an even distribution of points in position about the center of the response function. To minimize accelerations felt by the sample, the peaks of the triangle have quadratic accelerations and decelerations and are usually outside of the region of data collection. As a result of the architecture of instrument, the data gathered by the motor and SQUID control electronics are independent and are synchronized with a sync signal. The data is independently transmitted to the control software running on a computer and is combined as a function of time. The software then fits the voltage as a function of position to the response function for a point source moving through a second order gradiometer (Figure 4-4). The up and down scans are subtracted in position space, giving an approximation of the system drift over the time of the measurement. Linear drift is then subtracted from the SQUID signal as a function of time, and the raw data is fitted to the dipole response function in position space. The fitting algorithm assumes that the sample magnetic moment approximates a dipole magnetic moment, with sign and value remaining constant during measurement. Two fit calculations are made for each processed signal, one with the center position fixed and another with the center position as a free parameter. Both are shown in the lower panel of Figure 4-4.

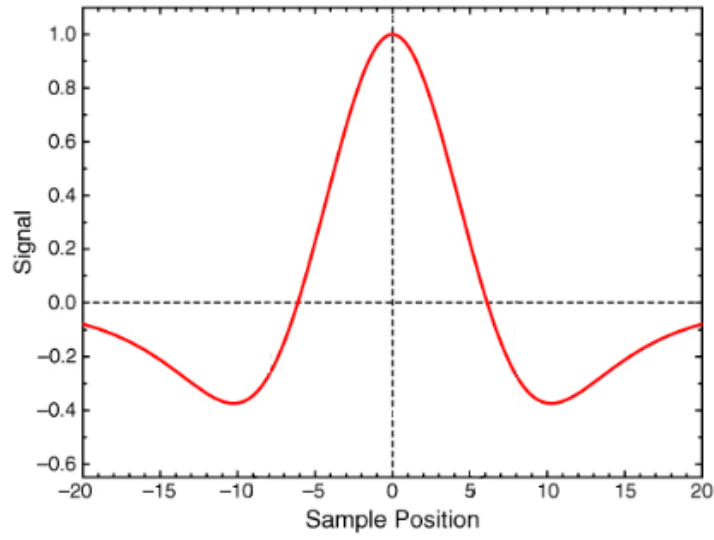


Figure 4-3. DC Scan response function.

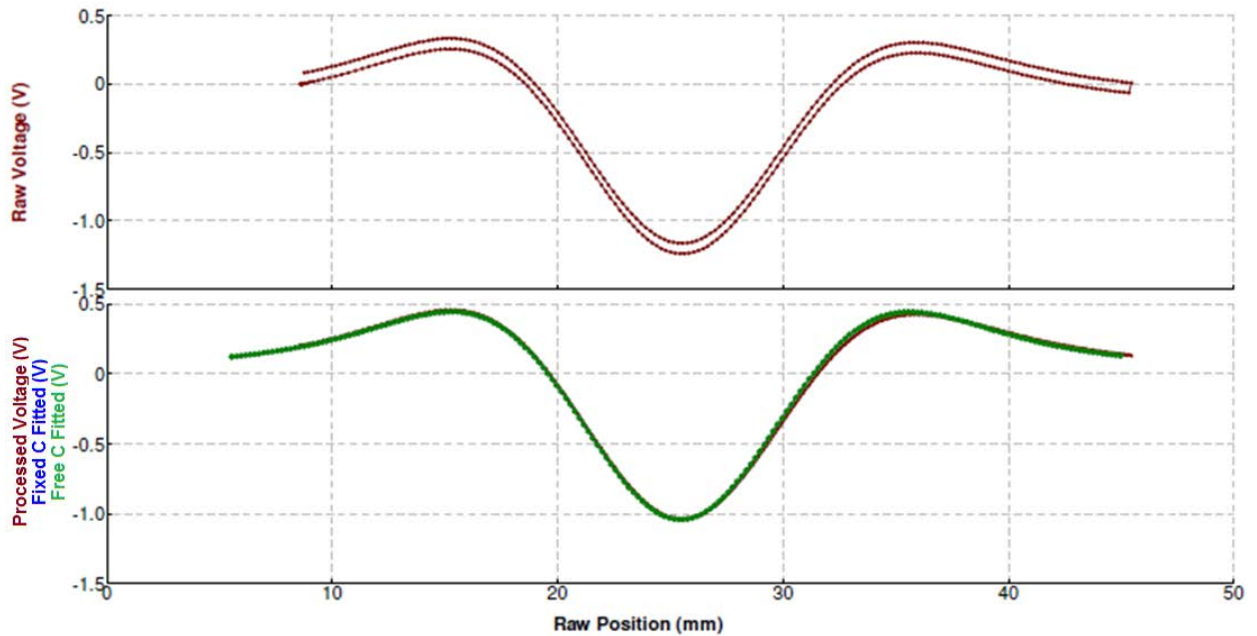


Figure 4-4. Voltage versus position curves, before and after fitting to the dipole response function.

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## 4.3 SQUID Drifts

Since there will be intrinsic DC drifts in the SQUID signal that result from various sources like magnet drift, sample position movement, etc. we need to ensure that our analysis of the signal is unaffected. During the DC scan, the measured SQUID response as function of position is fitted to an expected response function. By carefully selecting the starting point of each scan and executing both up and down scans using a linear motion with constant speed to collect the SQUID voltage as a function of position and time, we can subtract these two scans from each other in position space. The result will remove the sample signal from the scan and the remainder is the SQUID drift as a function of time and is equivalent to the intrinsic drift in the system. The drift is approximated as a linear function in time and removed from the data. Returning to position space, the raw data is fitted to a response function without having to worry about a linear contribution to the signal. This method is superior to earlier techniques, which add a linear term to the response function and fit that to the raw data. Unlike the older techniques that result in erroneous data fits if the data gathered for the response function is not perfectly centered or end effects introduce an asymmetric signal since a line and the response function are not orthogonal.

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## 4.4 Sample Geometry Effects

### 4.4.1. Scanning for Sample Offset

The MPMS3 allows for centering the sample using a DC scan along the entire length of the sample chamber smoothly. It fits the data to an expected waveform and calculates the center position. 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, the user should carefully consider whether the calculated sample offset is accurate or not.

### 4.4.2. Detection Coil Geometry

For reference, the dimensions of the MPMS 3 detection coils are 17 mm (diameter) and 8 mm (height, center to either outer coil). Refer to Figure 4-2. 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:

#### 4.4.2.1 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. 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. For most measurements therefore, the recommended sample length is  $< 5$  mm. This factor can be 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.

#### 4.4.2.2 FILLING FACTOR AND RADIUS EFFECTS (NARROW SAMPLES)

To a very good approximation, the lines of magnetic flux due to a sample will 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.

A common technique to determine the filling factor of a sample with a non-standard geometry is carefully measuring a reference sample 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.

#### 4.4.2.3 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 of 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 can be located in the references at the end of the chapter.

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## 4.5 Sample Holder Considerations

### 4.5.1 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.

## 4.5.2 Supplied Sample Holders

The sample holders supplied with the instrument are approximately 130 mm long, allowing the sample to be mounted  $66 \pm 3$  mm from the lower end. The sample transport 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.

## 4.5.3 Magnetic Impurities in the Sample Holder

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. The background of each sample holder should be measured prior to mounting samples.

## 4.5.4 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 move synchronously with the sample transport encoder array, then the reported moment by the instrument will be incorrect. 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.

## 4.5.5 End Effects

In an idealized measurement, the sample holder is made of a long, uniform material. Where this is the 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.

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# 4.6 Sample Insertion Considerations

## 4.6.1 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 and have no damage, like scratches, dents, etc.

## 4.6.2 Sample Exposure to Magnet Fields

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

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# 4.7 Sample Location Considerations

## 4.7.1 Angular Dependence

It is also important to mount the sample so that it moves smoothly on the axis of the sample rod. If the sample moves off-axis, the user 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.

## 4.7.2 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.

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# 4.8 Sequence Considerations

## 4.8.1 Moment Versus Field (M(H))

The MPMS 3 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 tend to result in scatter or imprecise data. Moreover, due to coupling of the magnet to the gradiometer, the SQUID will super-impose a linearly changing signal to the sample signal. This linear signal might be several orders of magnitude larger than the signal from the sample and can cause artifacts which vary with ramping speed and direction. This large linear signal will also cause the SQUID to reset often which limits the maximum ramping rate that can be used while measuring. Due to these issues, it is generally more advantageous to measure at stable fields rather than while sweeping in the MPMS 3.

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

## 4.8.2 Moment Versus Temperature ( $M(T)$ )

The MPMS 3 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 scatter or imprecise data, depending on the rate of temperature and moment change.

## 4.8.3 Moment Versus Time ( $M(t)$ )

The speed of the measurements makes it feasible to examine some time-dependent magnetic effects with the MPMS 3. However, the time required to stabilize the magnetic field with the QuickSwitch is on the order of 1 second, and the minimum time required to collect one data point (minimum practical averaging time) is about 1 sec, depending on the computer workload. Time-dependent effects faster than this cannot be resolved with the standard MPMS 3 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 remnant magnetization as a function of time, set the instrument magnetic field to zero using the fastest charging rate. If the instrument remnant 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 remnant magnetic field.

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## 4.9 Common Sources of Error

## 4.9.1 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 remnant 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 remnant 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 remnant field reproducible for a given measurement series. The remnant field will differ if you use different charging operations to set the same magnetic field. You may need to characterize the remnant magnetic field following all magnet charging operations in your sequence. Our experience clearly demonstrates the accuracy this procedure will achieve.

## 4.9.2 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.

## 4.9.3 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.

## 4.9.4 Loosely Mounted Samples

When samples are not mounted securely to the sample holder they will not undergo smooth motion within the detection coils. The sample signal will be rejected, 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.

## 4.9.5 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 ErYAG.

If there is a reason to assume that the thermometer calibration of the system is no longer valid, the calibration can be checked by looking at the superconducting transition temperature of indium and compare the result with the measurement performed during the original system installation.

The user kit contains an indium sample which should be measured using the “Temperature Accuracy, Oxygen Test” sequence located in the installation folder – the superconducting transition should occur at 3.40K +/-1%. Please contact your Quantum Design representative if this temperature verification indicates that the temperature calibration of the system is no longer correct.

## 4.9.6 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 DC Scan 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.

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## 4.10 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, 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, October 2006
- SQUID Sensors: Fundamentals, Fabrication and Applications, Series: NATO Science Series E, Vol. 329, Weinstock, H. (Ed.), ISBN-10: 0-7923-4350-6.



# Maintenance and Servicing

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## A.1 Overview

**In this appendix** we provide maintenance tips and instructions for user-serviceable items on your MPMS3. 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 and computer monitor.

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## 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.

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## 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 Removing a Loose Sample or Sample Holder from the Sample Chamber

Items that you will need for cleaning rod assembly:

- Double sided tape
  - Dental floss
- 1) In order to start this procedure the sample chamber should be at room temperature (300K).
  - 2) The field in the magnet should be set to 0 Oe.
  - 3) Place a small piece of double sided tape on the end of the cleaning rod assembly.
  - 4) To ensure that the tape does not fall off, tie a small amount of dental floss around the tape to secure the tape to the rod.
  - 5) The sample transport should be removed for this operation. Shut down MultiVu and remove the transport. Place the transport in the transport-shipping carrier.
  - 6) Insert the cleaning rod into the top of the sample chamber.
  - 7) Carefully push the rod down until it comes in contact with the item to be removed.
  - 8) Raise the rod up and slowly remove the rod from the chamber.
  - 9) Repeat as necessary to remove any debris from the chamber.
  - 10) If needed clean the chamber to insure no debris is left inside of the sample chamber.
  - 11) Replace the transport and restart MultiVu.

If the sample cannot be retrieved using the method described above, do not use hooks, screws, brushes, or springs on the end of the cleaning rod to try and retrieve the sample as doing so can damage the chamber wall. Contact a QD representative for assistance.

### A.3.2 Cleaning the Sample Chamber

Items that you will need for cleaning the rod assembly:

- Lint free wipes (Kimwipes®)
  - Isopropyl alcohol
- 1) Before you start this procedure the sample space should be at room temperature (300K) for at least 30 minutes.
  - 2) The field in the magnet should be set to 0 Oe.
  - 3) Remove any samples and sample rods.

- 4) Fold the Kimwipes® into a triangle (see figure A-1).
- 5) Insert a corner of the wipe in to the cleaning tip of the cleaning rod assembly.
- 6) Wrap the wipe around the tip of the cleaning rod.
- 7) Soak the wipe with alcohol.
- 8) Insert the tip of the cleaning rod into the top of the transport. The wipe should be able to go through the head without resistance - if it doesn't it should be re-wrapped a little tighter.
- 9) Insert the cleaning rod all the way into the sample chamber keeping the rod straight up and down.
- 10) Twist and move up and down to clean the sample chamber, turning in one direction as to not unravel the wipe from the cleaning tip.
- 11) Remove the cleaning rod by pulling out straight up.
- 12) Repeat several times until the wipe comes out clean.
- 13) After the sample chamber is clean set temperature to 320K and perform a purge-and-seal function.

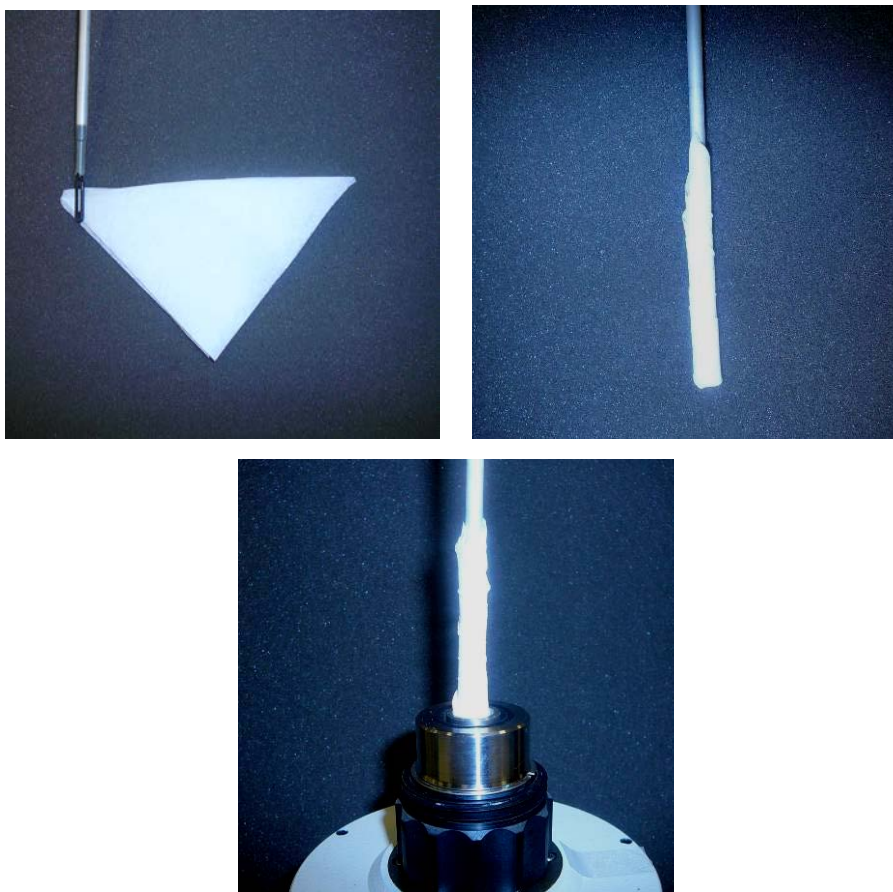


Figure A-1. Cleaning the sample chamber.



### A.3.3 Pump Oil, Changing and Filling

Only use the procedure outlined here if your MPMS 3 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. Loosen the screws on the side of the console. The front cover and sheet-metal enclosure 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:

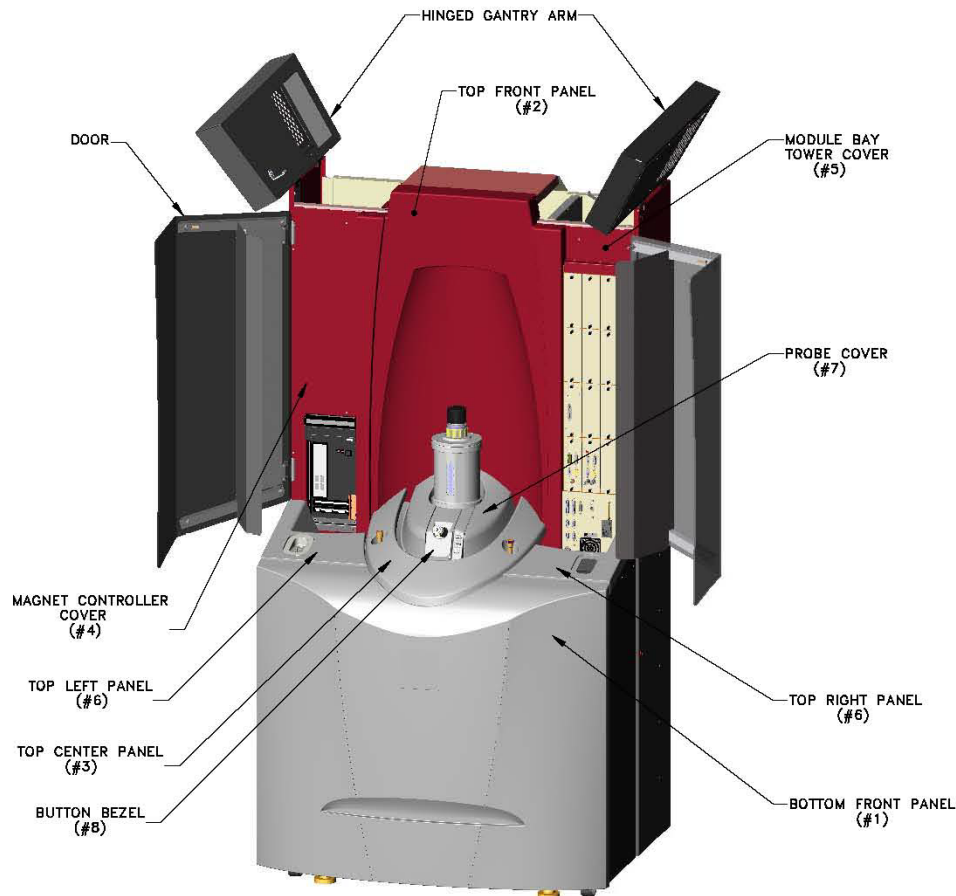


Figure A-2. Main cabinet cosmetic panels.

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 sample transport (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 Sample Transport Head: Removing and Reinstalling

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

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 sample transport. This operation will also ensure the sample chamber is set to room temperature before the sample rod is installed or removed. If no sample rod needed to be removed, set the temperature to 300 K and vent the sample chamber before proceeding.

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 sample transport by pulling straight out.
5. Make sure to have the sample transport 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 sample transport counter-clockwise while holding the sample transport. The transport is heavy and you will need to stabilize it.
7. Lift the sample transport straight up and place in the stand. **Never lay the sample transport on its side**—this can damage delicate suspension springs in the head.


To replace the Sample transport:

1. Position the sample transport 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 sample transport, 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 sample transport.
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) located at the four corners of the cabinet, 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.

	<b>WARNING!</b>
	Do not lift from the center of the dewar!
	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.

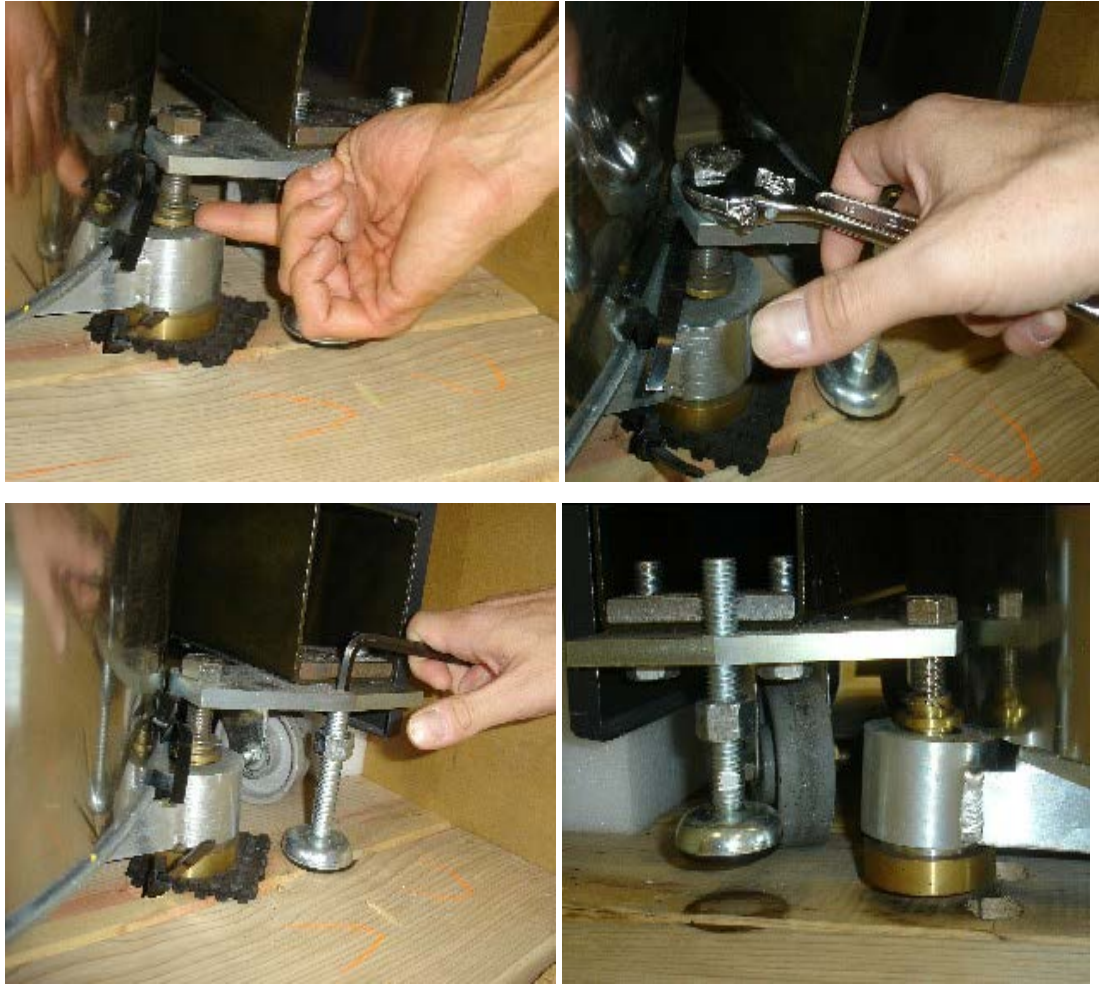


Figure A-3. Raising the leveling feet.

Adjust the large nut (clockwise) to raise it. Raise the steel feet using a 5/16 Allen Key. Turn counter-clockwise and raise them as high as the bottom of the feet with the large nut. 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 be 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 or Apiezon M 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. Service Note 1500-220 lists the commonly replaced O-rings, with pictures illustrating their locations. 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 assist in 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 to remove the magnet controller cover, as indicated in section A.3.4.

### A.3.10 Unplugging Nitrogen Exhaust

There are cases when the nitrogen exhaust mechanism of the MPMS 3 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 the same day. If both cryogenic liquids are filled in the same day it is recommended to perform both transfers simultaneously (starting with LN<sub>2</sub>) or waiting for 2-3 hours after LHe is filled before transferring LN<sub>2</sub>. 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. Refer to Chapter 1, section 1.5 for more details regarding filling the helium and nitrogen tanks.

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, situations could develop 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.

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## A.4 Initial Cooldown Procedure

The initial cooldown procedure is required after the system ran out of cryogenics and warmed up for an extended period of time. Typically at that stage the probe (including magnet) and dewar are at room temperature. For cooling down an MPMS 3 EverCool system, refer to the corresponding option manual.

**NOTE** If the MPMS 3 has run out of helium, but is cooler than room temperature, the standard helium fill utility should be used rather than the cooldown wizard.

Before starting the cooldown procedure you will need 100 liters of liquid helium, 100 liters of liquid nitrogen, a helium gas bottle with regulator, and the hose barb adapter for the helium fill port. 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 the pump and let it warm up for 5 minutes.

The cooldown wizard will prompt the user to verify that these steps have been completed prior to the start of the cooldown procedures.

### A.4.2 Starting the Cool Down Wizard

Start the cool down wizard to initiate the system cool down. Click the Helium Fill button on the instrument's front or select "Utilities > Helium Fill..." from the menu in MultiVu. Select "Yes" when asked whether this is an initial cool down to start the wizard.

**NOTE:** The cooldown wizard will only appear if the system has completely run out of helium and has warmed up. Otherwise, only the standard helium fill window will appear.

Closely follow the steps as outlined in the wizard to flush the system and probe and then cool down the system.

The wizard will first prompt you to connect the hose barb to the helium fill port on the port and attach the helium gas line. Next the wizard will prepare the dewar for the cooldown, by purging the dewar and then backfilling with helium (Figure A-4). Before each dewar pumpout or backfill procedure, the wizard will prompt you to close or open the helium gas supply valve, respectively (Figure A-5). Once the dewar preparation is complete, you have the option to perform the procedure again, simply by clicking "Start".

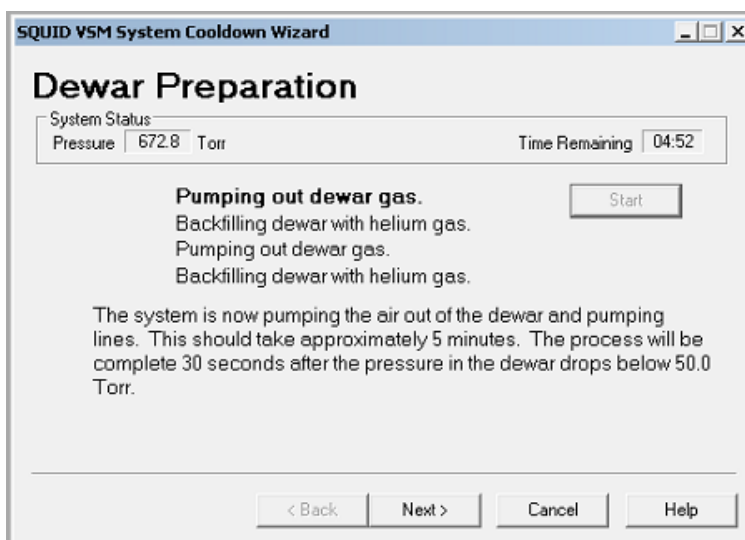


Figure A-4. Cooldown Wizard - Dewar Preparation Procedure



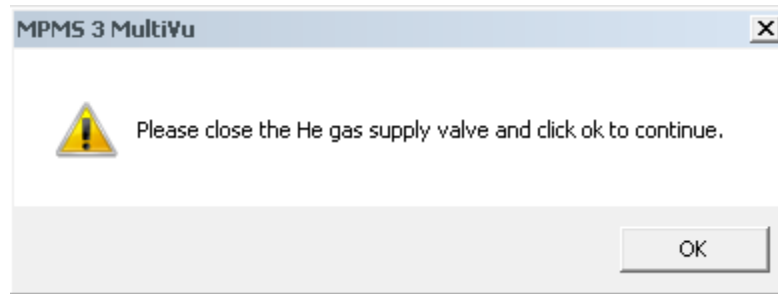


Figure A-5. Warning issued to close the helium supply valve prior to dewar pump-out

Annulus preparation is the next step in the cool-down wizard (Figure A-6). This portion of the wizard will check the CFE flow and verify that the proportional valve is operational.

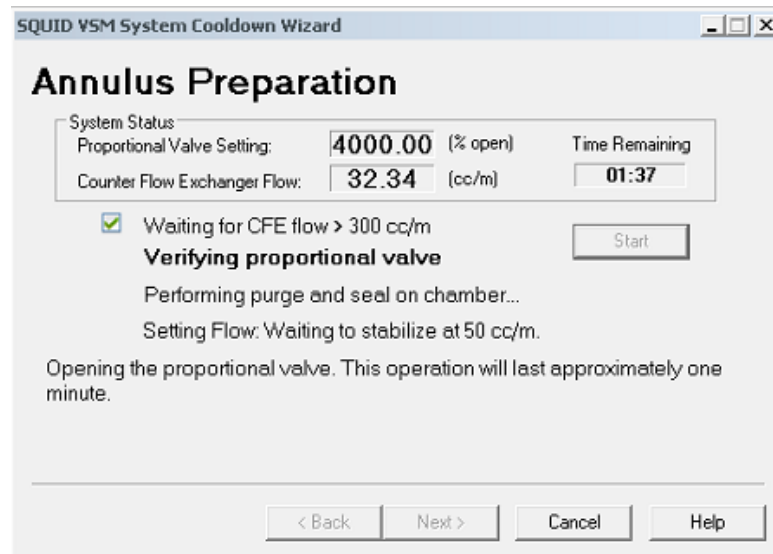
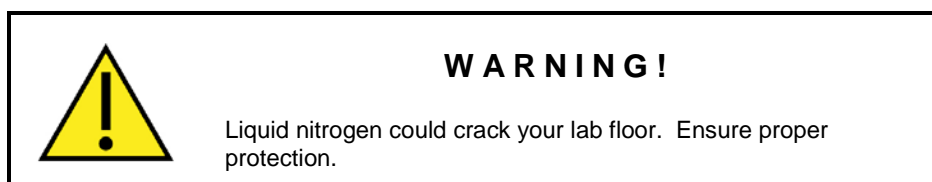


Figure A-6. Cool-down Wizard - Annulus Preparation Window

Once the flow has stabilized at 50 cc/min and the annulus preparation is complete, you will then be prompted to close the helium gas supply valve and remove the hose bard from the helium fill port. A dialog will also appear, instructing you to begin filling both liquid nitrogen and liquid helium. The filling of both cryogenes should be done simultaneously.

Fill LHe to about 60-80%. If the storage dewar runs dry stop immediately. The level meter is not operational for entire duration of the helium fill portion of the cool-down wizard, but instead is only on for brief intermittent periods. This is to avoid the extra heat load from the level meter. The progress of the cool-down will be shown by the green indicator on the last page of the cool-down wizard.

Fill LN2 until the tank is full (liquid nitrogen will spit out of the rubber exhaust hose).



### A.4.3 Verifying Probe is Working:

1. Close the cool down wizard when it has completed.
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.



# Advanced Software Operations

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## 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.

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## 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 ( $\Phi_0$ ) measured by the SQUID. As the output voltage from the SQUID electronics is in the range  $\pm 5V$  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-m<sup>2</sup>) 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.

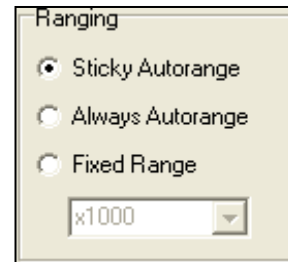


Figure B-1. SQUID ranging.

### 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.

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## 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.

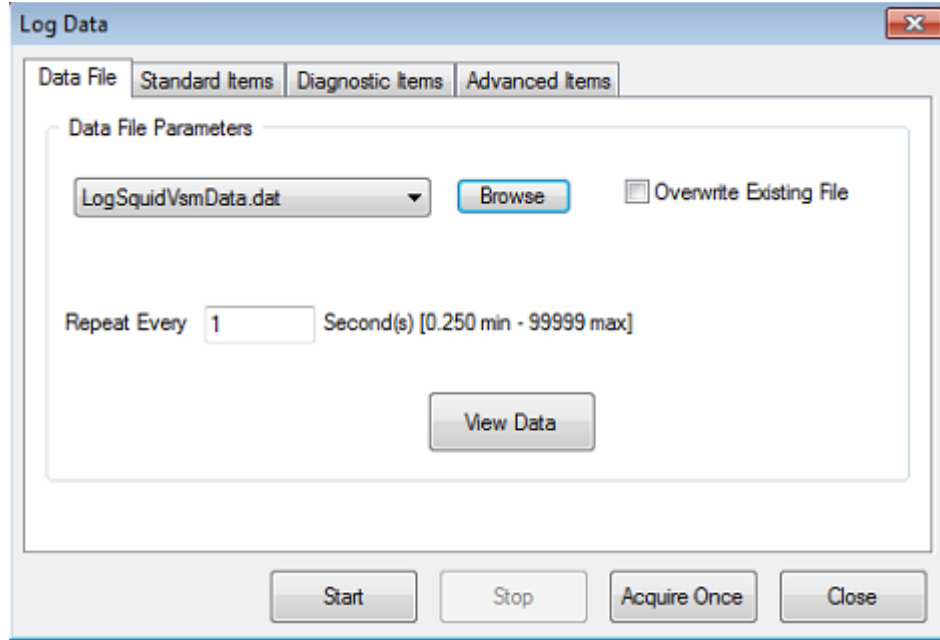


Figure B-2. Data logging interface.

### 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

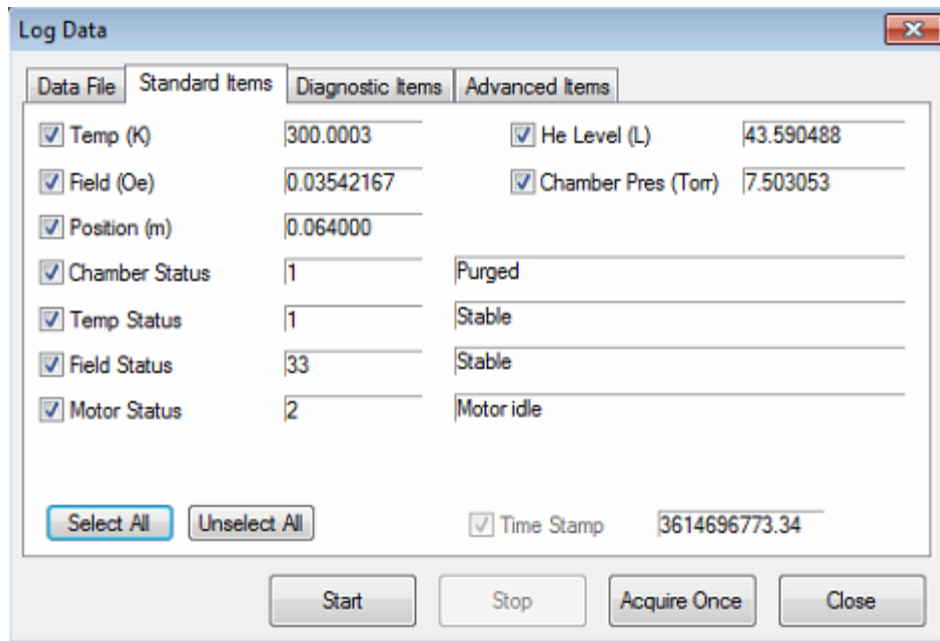


Figure B-3. Selection of standard data to log.

To learn the meaning of status codes that have been logged to a data file, open the status calculator dialog by selecting “Utilities > Diagnostic > Status Calculator” from the menu bar. The status codes for temperature control, field control, and sample chamber can be calculated, as well as the time stamp.

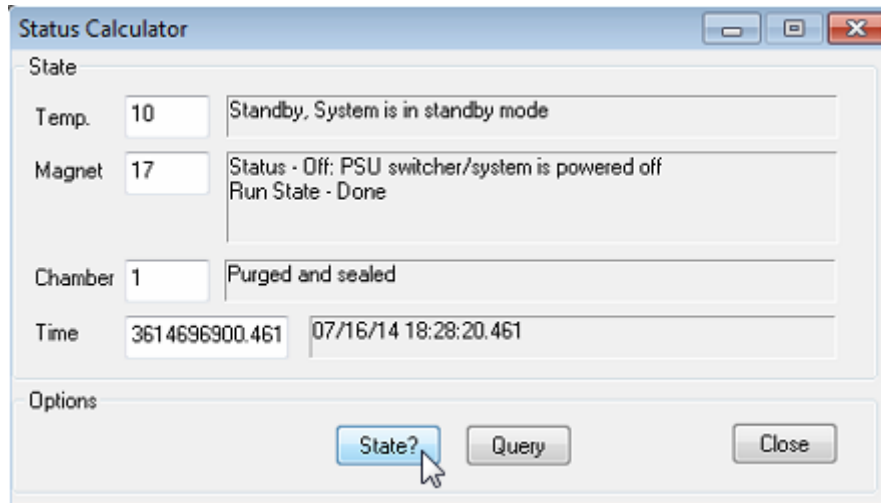


Figure B-4. Status Calculator

### 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 Qmap 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.

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## 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 MPMS3, 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.

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## B.5 SQUID Diagnostics

MPMS 3 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 ( $V/\Phi_0$ ). 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.”

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## B.6 Resetting the Magnet

Because it poses no resistance to the motion of electrons, the superconducting solenoid in the MPMS 3 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 gradients 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.

# System Specifications and Interconnects

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## C.1 Overview

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

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## C.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.

### C.2.1 Power Ratings



#### **WARNING!**

Voltage: 200-230 VAC, 50/60 Hz, single phase  
Current: typical operation 6-8 A



#### **WARNING!**

Mains Fusing: 10 A breaker at main power switch.

## C.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

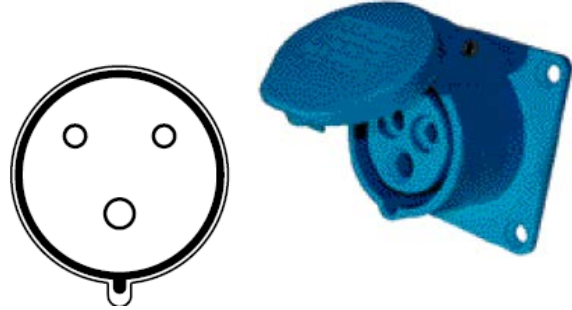


Figure C-1. Laboratory power outlet.

## C.2.3 Laboratory Ambient Conditions

Operating ambient temperature: 10-40 °C (50-104 °F)

Operating ambient relative humidity: 10-90% (non-condensing)

## C.2.4 Cryogenic Requirements

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

Liquid Nitrogen: 100 liters

Liquid Helium: 100 liters (another 100 liters should be available as a back-up)

Pressurized Helium gas cylinder with regulator (regulator pressure range: 100-300 mbar)

Under normal operating conditions the cryogen hold-times are typically 12 days for helium (100% = 70 liters) and nitrogen (100% = 55 liters). See section 1.5 for more details on transferring cryogenes. For EverCool system configuration, please refer to the corresponding power and cryogenic requirements.

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

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## C.3 System Interconnects

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

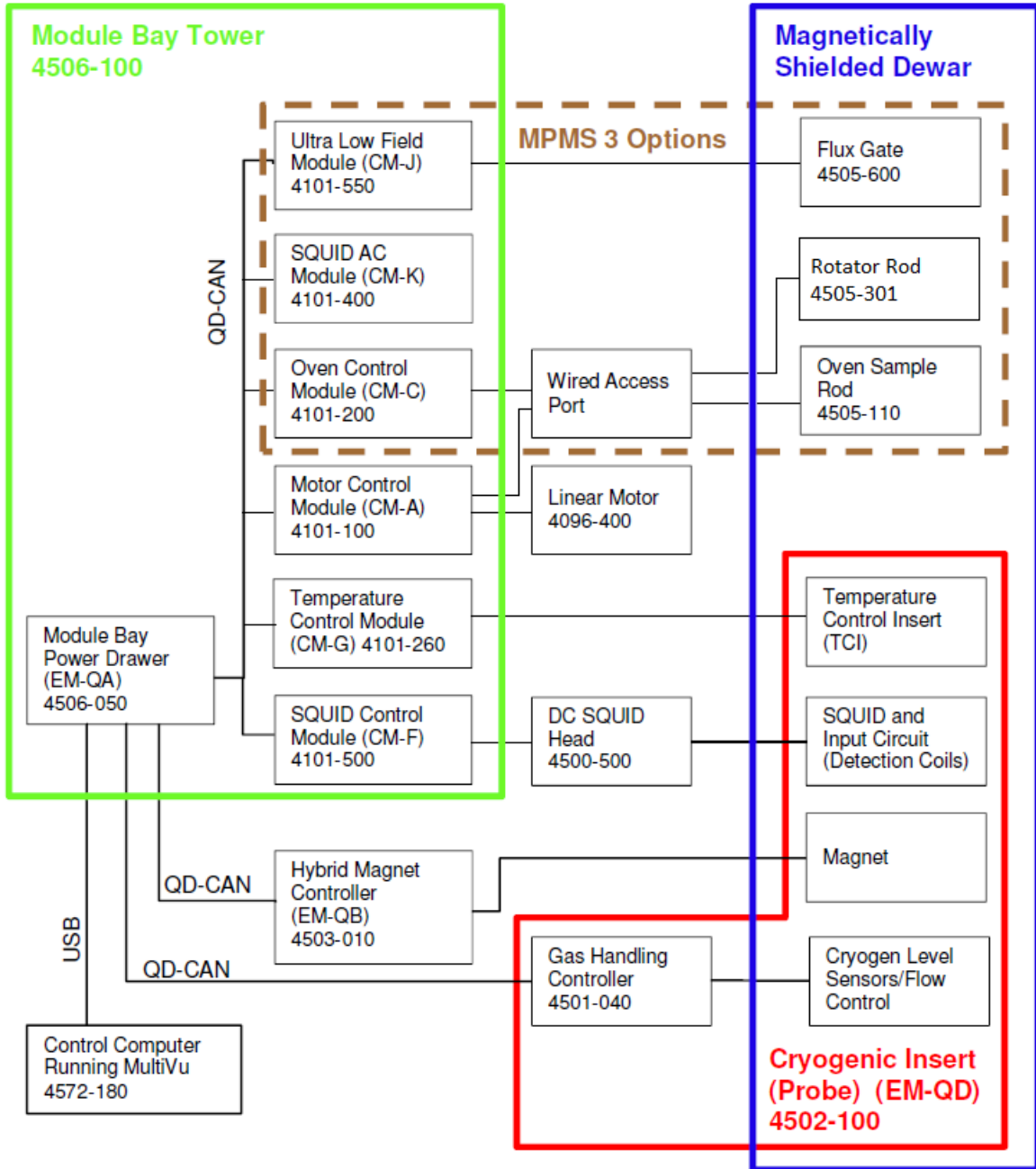


Figure C-2. MPMS 3 System Architecture

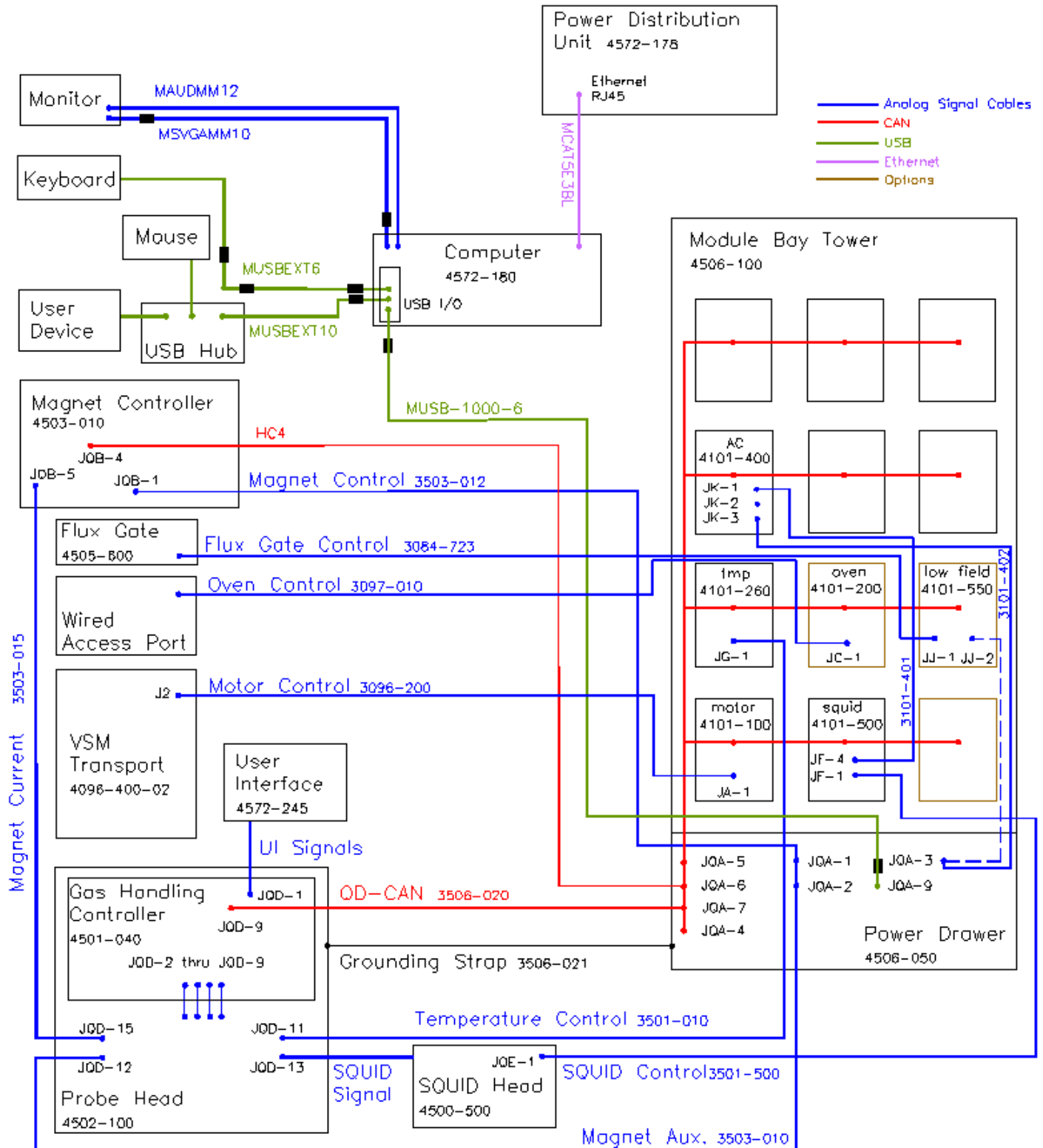


Figure C-3. MPMS 3 System Interconnects.

### C.3.1 Master List of Connections

A master list of connections is provided below for the standard MPMS 3 modules and controllers as well as option modules.

Table C-1. Connections List – SQUID Control Module

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
SQUID CONTROL: 4101-500, JF1/2 (CH1/2)	SQUID CONTROL: 3501-500	SQUID HEAD: 4500-500, JQE-1
1	DETECTOR A	1
9	DETECTOR B	9
2	SIGNAL A	2
10	SIGNAL B	10
3	+15 VOLTS	3
11	-15 VOLTS	11
4	MOD/BIAS A	4
12	MOD/BIAS B	12
5	TEST INPUT	5
13	REF GND	13
6	RANGE/HEATER/RESET	6
14	RANGE/HEATER/RESET	14
7	EXT INPUT A	7
15	EXT INPUT B	15
8	SHIELD	8
SQUID CONTROL: 4101-500, JF-4 (AUX)	SVSM AC-SQUID MODULE: 3101-404	SQUID AC: 4101-400, JK-1 (SQUID AUX)
3	SIGNAL A	15
8	SIGNAL B	8
1	FEEDBACK +	1
6	FEEDBACK -	9
5	RESET	5

Table C-2. Connections List – SQUID Head Module

<b>MODULE/PART #/CHANNEL</b>	<b>CABLE ASSEMBLY</b>	<b>MODULE/PART #/CHANNEL</b>
<b>PIN #</b>	<b>FUNCTION</b>	<b>PIN #</b>
SQUID HEAD: 4500-500, PIGTAIL	MPMS3 FLEXIBLE DC SQUID: 3502-050	SQUID CAPSULE ASSY.: 4502-001
E11	FEEDBACK A	1
E12	FEEDBACK B	2
E7	MODULATION A	3
E8	MODULATION B	4
E3	SIGNAL A	5
E4	SIGNAL B	6
E13	SQUID HEATER A	7
E14	SQUID HEATER B	8
E5	BIAS A	9
E6	BIAS B	10
E9	INPUT TRSF HEATER A	11
E10	INPUT TRSF HEATER B	12
E2	SHIELD/GRD	13

Table C-3. Connections List – Temperature Control Module

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
TEMPERATURE CONTROL MODULE: 4101-260, JG-1	TEMPERATURE CONTROL: 3501-010	SAMPLE TUBE: 4501-500, JQD-11
1	GAS HEATER THERMOMETER I+	1
16	GAS HEATER THERMOMETER I-	16
2	GAS HEATER THERMOMETER V+	2
17	GAS HEATER THERMOMETER V-	17
3	SAMPLE THERMOMETER I+	3
18	SAMPLE THERMOMETER I-	18
4	SAMPLE THERMOMETER V+	4
19	SAMPLE THERMOMETER V-	19
5	NECK THERMOMETER I+	5
20	NECK THERMOMETER I-	20
6	NECK THERMOMETER V+	6
21	NECK THERMOMETER V-	21
7	SPARE THERMOMETER I+ (NOT USED)	7
22	SPARE THERMOMETER I- (NOT USED)	22
8	SPARE THERMOMETER V+ (NOT USED)	8
23	SPARE THERMOMETER V- (NOT USED)	23
12	SROM GRD	12
44	SROM SI	44
13	SROM SO	13
14	SROM CS	14
15	SROM VCC	15
30	SROM SCK	30
31	GAS HEATER I+	31
32	GAS HEATER I-	32
33	SAMPLE HETER I+	33
34	SAMPLE HETER I-	34
35	NECK HEATER I+	35
36	NECK HEATER I-	36
37	SPARE HEATER I+ (NOT USED)	37
38	SPARE HEATER I- (NOT USED)	38



Table C-4. Connections List – Ultra Low Field Module

<b>MODULE/PART #/CHANNEL</b>	<b>CABLE ASSEMBLY</b>	<b>MODULE/PART #/CHANNEL</b>
<b>PIN #</b>	<b>FUNCTION</b>	<b>PIN #</b>
MPMS 3 ULF: 4101-550, JJ-2 (MOD TRIM)	SVSM AC MOD/TRIM COIL: 3101-402	POWER DRAWER ASSEMBLY: 4506-050, JQA-3 (MOD COIL)
8	MOD +	13
4	MOD -	5
9	TRIM +	12
5	TRIM -	4
MPMS 3 ULF: 4101-550, JJ-1 (FLUXGATE)	SVSM FLUXGATE: 3084-723	FLUXGATE FISCHER CONNECTOR
1	INTEGRATOR RESET A	1
6	INTEGRATOR RESET B	2
2	SENSOR DETECT A	3
7	SENSOR DETECT B	4

Table C-5. Connections List – SQUID AC Module

<b>MODULE/PART #/CHANNEL</b>	<b>CABLE ASSEMBLY</b>	<b>MODULE/PART #/CHANNEL</b>
<b>PIN #</b>	<b>FUNCTION</b>	<b>PIN #</b>
SQUID AC: 4101-400, JK-1 (SQUID AUX)	SVSM AC-SQUID MODULE: 3101-404	SQUID CONTROL: 4101-500, JF-4 (AUX)
15	SIGNAL A	3
8	SIGNAL B	8
1	FEEDBACK +	1
9	FEEDBACK -	6
5	RESET	5
SQUID AC: 4101-400, JK-2 (CAL)	SVSM AC CAL/DETECT COIL: 3101-403	WAP FISCHER CONNECTOR -- > CAL COIL
2	CAL COIL +	4 --> 1
7	CAL COIL -	6 --> 2
SQUID AC: 4101-400, JK-3 (MOD TRIM)	SVSM AC MOD/TRIM COIL: 3101-402	POWER DRAWER ASSEMBLY: 4506-050, JQA-3 (MOD COIL)
8	MOD +	13
4	MOD -	5
9	TRIM +	12
5	TRIM -	4

Table C-6. Connections List – Motor Module

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
MOTOR MODULE: 4101-100, JA-1 (SERVO)	VSM, DRIVE (SVSM): 3096-200-02	VSM TRANSPORT: 4096- 400-02
	MOTOR -	14
	MOTOR +	12
1	MOTOR -	15
10	MOTOR +	13
2	+ 5 V	16
11	GND	4
19	SDO MOS 1	7
24	SPARE 1	10
3	+ A	17
12	- A	18
20	SDI MISO	8
23	AUXCS	2
4	+ B	5
13	- B	6
21	SCK	1
22	ROMCS	9
25	SPARE 2	3
26	SPARE 3	11
5	+ Z	N/C
14	- Z	N/C
8	+ LIM	N/C
17	+ LIMRTN	N/C
9	- LIM	N/C
18	- LIMRTN	N/C

With Rev D0 or higher, Motor Modules have rotator connection (see table C-7).

Table C-7. Connections list – Motor Module Stepper

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL	
PIN #	FUNCTION	PIN #	
MOTOR MODULE: 4101-100, JA-Z (STEPPER)	Rotator Cable: 3505-300	Feedthrough 4505-305 Rotator Rod	
		Fischer Connector	Omnetics Connector
1	MOTOR A+	5	1
9	MOTOR A-	4	2
2	MOTOR B+	12	3
10	MOTOR B-	6	4
3	VCC	11	7
11	GND	9	12
7	Limit +	7	8
15	Limit -	10	10
8	Limit RTN	8	11

Table C-8. Connections List - Gas Handling Module

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL	
PIN #	FUNCTION	PIN #	
POWER DRAWER ASSEMBLY: 4506-050,QD CAN, JQA-7	GAS HANDING, QD CAN: 3506-020	MPMS3 GHC: 4501-040GAS CONTROL, JQD-9	
7	CAN H	7	
2	CAN L	2	
8	SYNC H/RS	8	
4	SYNC L	4	
5	LINE SYNC	5	
6	SYS GND	6	
1	-24 VDC	1	
3	24V RTN	3	
9	+24 VDC	9	

Table C 9. Connections List - Oven Module

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
OVEN MODULE: 4101-200, JC-1	OVEN CONTROL: 3097-010	WAP FISCHER CONNECTOR -- > HEATER STICK
9	TC-NEG	6
1	TC-POS	4
2	THM-A	3
10	THM-B	5
7	HTR A	1
15	HTR B	2

Table C-10. Connections List - Magnet Control

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
POWER DRAWER ASSEMBLY: 4506-050, JQA-2 (MAG AUX OUT)	MPMS3, MAGNET CONTROL: 3503-010	PROBE HEAD, 4502-100, JQD-12
8	MAG VOLTAGE A	8
21	MAG VOLTAGE B	9
9	MOD COIL A	10
22	MOD COIL B	11
10	SWITCH HEATER A	12
23	SWITCH HEATER B	13
11	QUENCH HEATER A	14
24	QUENCH HEATER B	15
25	TRIM COIL A	16
13	TRIM COIL B	17
POWER DRAWER ASSEMBLY: 4506-050, JQA-1 (MAG AUX IN)	MPMS3 MAGNET CONTROL INTERFACE: 3503-012	HYBRID MAGNET CONTROLLER: 4503-010-01, JQB-1 (MAGNET CTRL)
8	MAG VOLTAGE A	8
21	MAG VOLTAGE B	21
10	SWITCH HEATER A	10
23	SWITCH HEATER B	23
11	QUENCH HEATER A	11
24	QUENCH HEATER B	24
HYBRID MAGNET CONTROLLER: 4503-010-01, JQB-5	MAGNET CURRENT: 3503-015	PROBE HEAD, 4502-100, JQD-15

1	CURRENT POS	1
2	CURRENT NEG	2

Table C-11. Connections List - Evercool Option

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
MANIFOLD: 4099-605, SV1	SOLENOID VALVE, EC: 3101-363; RECIRC VALVE	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-1
1	+24 V	1
2	GND	2
MANIFOLD: 4099-605, SV2	SOLENOID VALVE, EC: 3101-363; EXHAUST VALVE	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-2
1	+24 V	1
2	GND	2
MANIFOLD: 4099-605, SV4	SOLENOID VALVE, EC: 3101-363; SUPPLY VALVE	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-4
1	+24 V	1
2	GND	2
MANIFOLD: 4099-605, LP1	PRESSURE SENSOR: 3099-617; DEWAR PRESSURE	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-9
1	+12 V	1
2	GND	2
4	SIGNAL	3
MANIFOLD: 4099-605, LP2	PRESSURE SENSOR: 3099-617; DEWAR PRESSURE	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-10
1	+12 V	1
2	GND	2
4	SIGNAL	3
MANIFOLD: 4099-605, LP3	PRESSURE SENSOR: 3099-617; SUPPLY PRESSURE	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-11
1	+12 V	1
2	GND	2
4	SIGNAL	3
FAN, JQL-14	FAN 24 V: 3099-617	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-14
1	+24 V	1

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
3	GND	2
4 STAGE DC DIAPHRAM PUMP: 4095-306, P19	ADAPTER DC PUMP: 3095-243	BOX; EVERCOOL 2 CONTROLLER: 4099-641, JQL-18
1	SWITCH COMMON (K1-COM)	1
2	CNTROL (K1-N.O.)	2
3	PUMP SPEED V	3
4	GND	4
5	+24 V	5
6	GND	6
EVERCOOL-2 CONTROLLER: 4099-600, JQL-8	INTERLOCK CABLE: 3099-625-01, -02	CRYOMECH COMPRESSOR: 4099-610 OR -611, -612, SYSTEM I/O
1	+24 V	10
2	GND	13
N/C		9
N/C		11
EVERCOOL-2 CONTROLLER: 4099-600, JQL-17	SEARIAL CABLE: 3099-624-01, 02	CRYOMECH COMPRESSOR: 4099-610 OR -611, -612, RS232/485
1	DSR	N/C
2	CD	N/C
3	DTR	N/C
4	GND	5
5	RX	2
6	TX	3
7	CTS	N/C
8	RTS	N/C
EVERCOOL-2 CONTROLLER: 4099-600, JQL-13	COLD HEAD: 3099-627	PULSE TUBE COLD HEAD: 4099-608
5	CABLE SHIELD	N/C
2	THERMOMETER I+	7
1	THERMOMETER I-	8
3	THERMOMETER V+	9
4	THERMOMETER V-	10
6	HEATER +	1

MODULE/PART #/CHANNEL	CABLE ASSEMBLY	MODULE/PART #/CHANNEL
PIN #	FUNCTION	PIN #
7	HEATER -	2

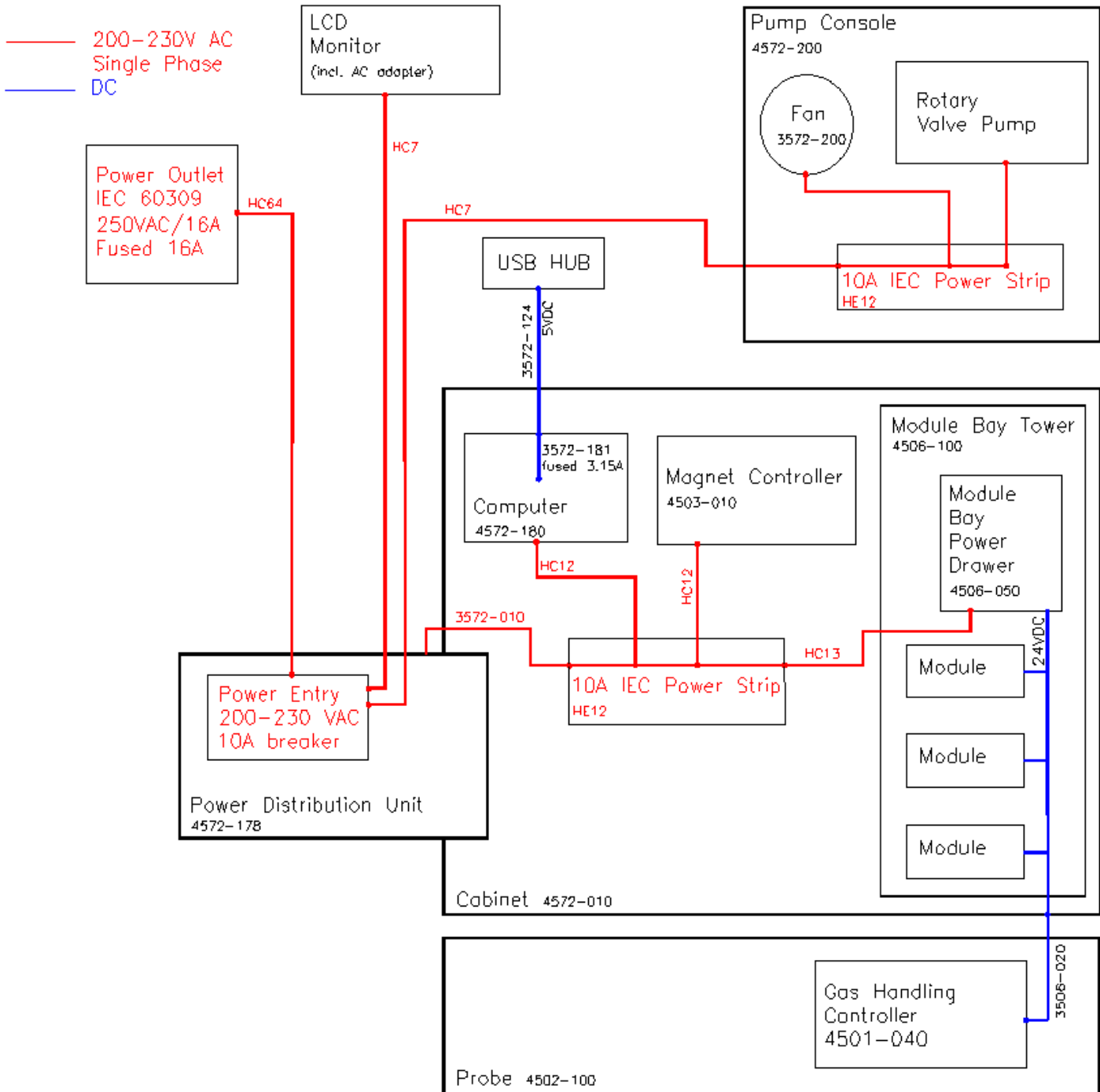


Figure C-4. MPMS 3 Power Distribution Diagram

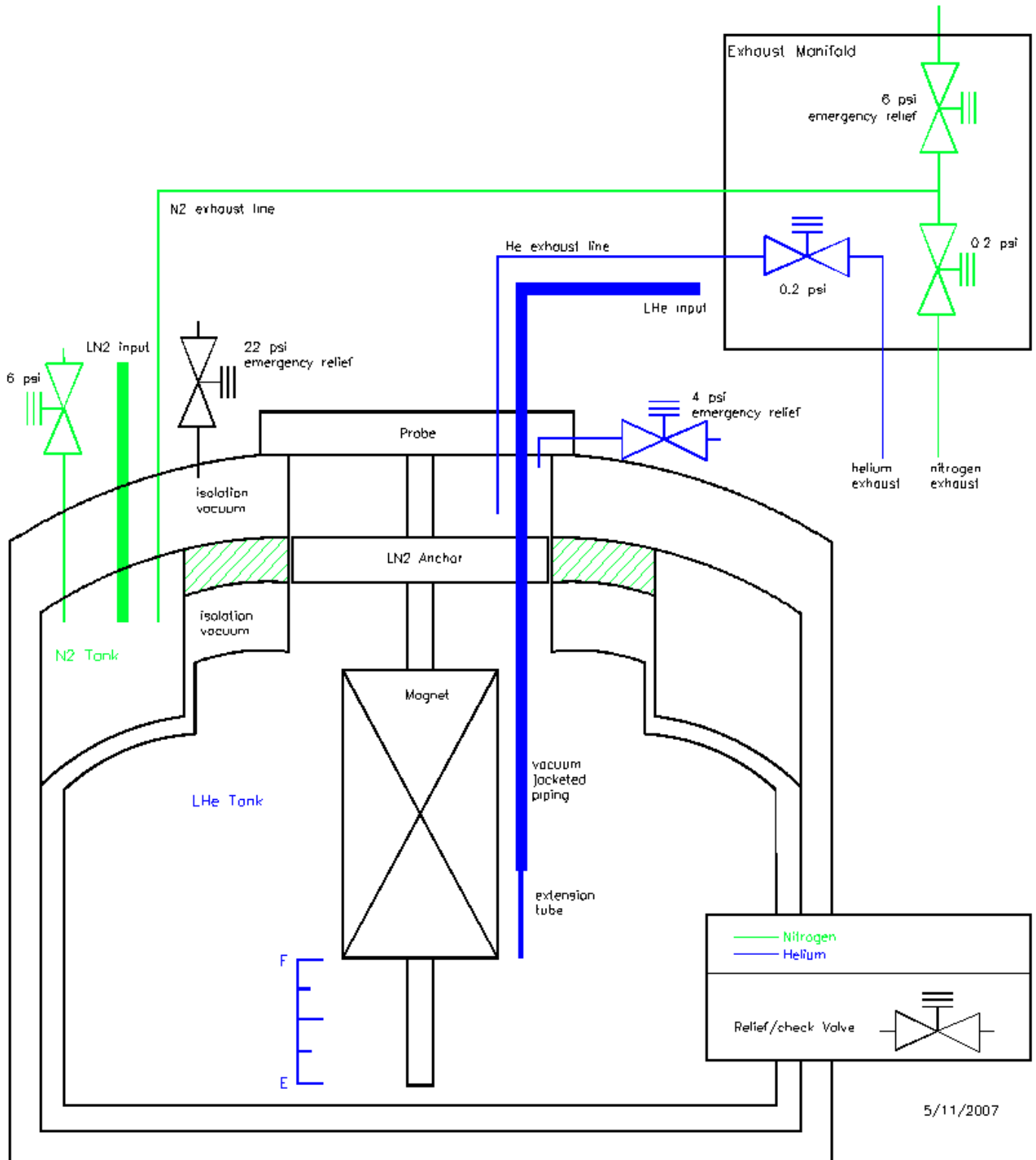



Figure C-5. MPMS 3 Cryogen Plumbing Diagram



### C.3.2 Attaching a Printer to the System

The recommended solution is to print through a network printer. If this is not available or possible, a printer can be attached to the system. Due to noise issues and possible ground loops it is required when hooking a printer up to the system, the power be plugged into the pump cabinet and the USB cable be plugged into the marked USB outlet on the computer or the provided USB hub.

	<b>WARNING!</b>
The power provided on the power strip for the pump cabinet is 220V.	

Please contact a Quantum Design representative if you have any questions or concerns about attaching a printer to the system.

# Understanding the Data Files Format

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## D.1 Overview

In this appendix the meaning of each data column in a measurement data file generated by the MPMS3 system is explained in some details.

## D.2 Data File Columns Descriptions

Table D-1. Standard Measurement File Data Columns

COLUMN	DESCRIPTION
<b>General Items</b>	
Comment	Any comments entered into the data file. Comments can be entered either directly using the Measure → Datafile Comment... menu entry or inside a sequence using the Measurement Commands → VSM → Datafile Comment sequence command.
Time Stamp (sec)	The absolute time stamp for the current row of data. Timestamps can be converted into date & time values using the Utilities → Status Calculator menu item in MultiVu (enter the timestamp from the data file into the Time text field and click State? to convert the value).
Temperature (K)	The sample temperature for the current data row. This is the sample temperature, taking into account thermal history and current measurement options (e.g., it might be the temperature of the oven sample holder when performing measurements with the oven option active).

COLUMN	DESCRIPTION
Magnetic Field (Oe)	<p>The magnetic field for the current data row.</p> <p>This value takes into account current measurement options (e.g., field reported by the Ultra-Low Field Option).</p>
<b>DC Scan Related Items</b>	
DC Moment Fixed Ctr <sup>Error! Bookmark not defined.</sup> DC Moment Err Fixed Ctr <sup>Error! Bookmark not defined.</sup>	<p>Amplitude of the moment and associated standard error for DC scan measurements (fixed center)</p> <p>The “Fixed Center” values denote the analysis result where the sample position is determined by the AutoTracking algorithm, the moment thus being the only free parameter for the fit to the raw data.</p>
DC Moment Free Ctr <sup>Error! Bookmark not defined.</sup> DC Moment Err Free Ctr <sup>Error! Bookmark not defined.</sup>	<p>Amplitude of the moment and associated standard error for DC scan measurements (free center)</p> <p>The “Free Center” values denote the analysis result where both, the sample location and the sample moment, are free parameters for the fit to the raw data.</p>
DC Fixed Fit DC Free Fit	<p>Quality of fit of the raw data to the dipole response function for the “Fixed Center” and “Free Center” fits, respectively</p> <p>Values range from 0 to 1 with 1 being a perfect fit and 0 being random data with no correlation to the expected response function</p>
DC Calculated Center (mm) DC Calculated Center Err	<p>Calculated sample position and associated estimated error for the “Free Center” fit to the DC scan raw data</p> <p>This value uses the same coordinate system as the “Center Position (mm)” value – for perfectly centered samples and clean signals those two values should match within the resolution of the transport encoder.</p>
DC Scan Length (mm)	Scan length of the current data point (as selected by the user in the “Measure” menu)
DC Scan Time (s)	Scan time of the current data point (as selected by user in the “Measure” menu)
DC Number of Points	Number of points in the raw DC scan waveform
DC SQUID Drift	SQUID drift calculated from the subtraction of the up and down measurement scans
DC Min V (V) DC Max V (V)	<p>Maximum and minimum voltage reported by the SQUID module during the DC scan.</p> <p>These values represent the uncorrected values without taking into account drifts. Values are reported as raw voltage multiplied by the SQUID range as reported in the next column (i.e., maximum reported value can be as high as <math>\pm 5000</math> in range 1000).</p>
Range	<p>Range setting of the SQUID module for the current data point.</p> <p>Possible values are 1, 10, 100, and 1000 (with 1 being the most sensitive setting).</p>
<b>Diagnostic Items</b>	

COLUMN	DESCRIPTION
Min. Temperature (K) Max. Temperature (K)	Minimum and maximum sample temperature readings over the time required to measure the current data point.  These values are especially useful when measuring while sweeping temperature as they give an indication about the temperature accuracy for the data point.
Min. Field (Oe) Max. Field (Oe)	Minimum and maximum sample field readings over the time required to measure the current data point.
Mass (grams)	Total mass of moving parts obtained from the DC component of the motor force.  This includes the armature inside the linear motor, the sample rod, sample holder, and the sample itself.
Motor Lag (deg)	Phase lag between motor drive current and actual motor position.
Pressure (Torr)	Pressure inside the sample chamber for the current data point.
Measure Count	Total number of waveforms used to calculate the current data point.  This number indicates the quality of the data point – in perfect conditions, it should be ( <i>Frequency x Averaging Time</i> ) but can be reduced due to waveform rejection inside the SQUID module (e.g., SQUID resets occurring during the measurement).  If the Measure Count drops below 50% of the expected value, the data point will be rejected and not logged in the data file.
Measurement Number	Measurement repetition number for MvsH and MvsT measurements.
SQUID Status (code)	Internal status codes as reported by the SQUID module.
Motor Status (code)	Internal status codes as reported by the motor module.
Measure Status (code)	0                   OK 1                   SQUID voltage railed
Motor Current (amps)	AC component of the motor current.  Value is proportional to the force required to achieve the requested vibration amplitude.
Motor Temp. (C)	Temperature of the heat sink inside the motor module.  This temperature is used internally from the motor module to prevent damage to the module and the motor. If the temperature exceeds a maximum value, the motor will stop moving and an error will be logged into the event log.
Temp. Status (code)	Temperature status code as reported by the temperature control subsystem.  Status codes can be translated into corresponding status text using the Utilities → Status Calculator... menu entry in MultiVu (enter the status code from the data file into the Temp. text field and click State? to convert the value).

COLUMN	DESCRIPTION
Field Status (code)	<p>Field status code as reported by the magnet power supply.</p> <p>Status codes can be translated into corresponding status text using the "Utilities → Status Calculator..." menu entry in MultiVu (enter the status code from the data file into the Magnet text field and click State? to convert the value).</p>
Chamber Status (code)	<p>Chamber status code as reported by the Gas handling controller.</p> <p>Status codes can be translated into corresponding status text using the "Utilities → Status Calculator..." menu entry in MultiVu (enter the status code from the data file into the Chamber text field and click State? to convert the value).</p>
Chamber Temp (K)	<p>Chamber temperature for the current data point.</p> <p>This value is the instantaneous temperature reported by the chamber thermometer on the sample tube and will be different from the sample temperature reported earlier as this does not take thermal history into account.</p> <p>When the oven option is active, the chamber temperature should be stable at about 280 K and can be used to diagnose oven performance issues.</p>
Redirection State	<p>0                      No redirection 1                      Oven option active and controlling temperature</p>
Map 01 ... Map 16	<p>Mappable data columns (varying content).</p> <p>Additional data columns available for logging advanced diagnostic data (see section B.3.3 in the SVSM User Manual). Actual column titles in the data file will reflect the data being recorded when the data file was first created.</p>

# Using the MPMS 3 Superconducting Magnet at Low Fields

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## E.1 Overview

The superconducting magnet used in MPMS 3 is capable of generating fields up to 7 tesla ( $7 \times 10^4$  gauss) with the value determined to a very high degree of accuracy over most of this range. The magnetic field value reported to the user is based only on the current flowing from the magnet power supply. At low fields (less than 1 tesla), the magnetic field experienced by the user's sample can deviate significantly from the reported magnetic field. The magnitude of this field error can be as much as 40 gauss in MPMS 3 magnets. The origin of this effect is described and methods for its mitigation are outlined.

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## E.2 Introduction

When using a superconducting solenoid to control the magnetic field in an experiment, the user must be aware of pinned magnetic flux lines and flux movement within the magnet in order to better know the magnetic field at the location of the user's experiment. This appendix will discuss how these effects manifest in the magnet used for the Quantum Design MPMS 3. As the title implies, the effects are of greatest relative significance at low fields and are very important to keep in mind when conducting research on soft magnetic materials whose magnetic state is strongly affected by small changes in magnetic fields near zero.

Quantum Design does not incorporate a field sensor such as a Hall sensor in its instruments. The magnetic field reported by the MPMS 3 is based on the current that is being generated in the magnet power supply (if the magnet is charging), or the last current that was sourced from the power supply when it was connected to the magnet (if the QuickSwitch on the magnet has been closed). The magnet power supply current is translated into a magnetic field using a constant field-to-current conversion factor (or "B/I ratio") for that given magnet. This yields a magnetic field value that is very accurate at higher fields (above about 1 tesla).

There are two phenomena that cause the MPMS3 reported magnetic field to differ from the actual magnetic field at the location of the user's sample. Below is a brief summary of these effects. The remainder of this appendix will describe each of these phenomena in greater detail.

- 1) **Magnet remanence:** alloys such as those used in the windings of superconducting magnets will permit the magnetic field to enter the superconducting material as threads of quantized magnetic flux when placed in sufficiently large magnetic fields. These flux lines are "pinned" and immobilized at defects in the material. Movement of flux lines causes energy dissipation and can destroy the superconducting

state. When the magnetic field is removed (for our case, when the magnet current is set back to zero from a high field), some of these pinned flux lines remain behind and create a small magnetic field seen at the sample location.

- 2) **Flux creep and escape:** over time, some of these pinned magnetic flux lines will redistribute and may even leave the wire material altogether. The term “flux creep” describes the diffusion of flux lines inside a superconductor. At a stable magnetic field, escape of flux from the interior of the wire can lead to induced currents in the magnet due to the topology of the superconducting magnet.

Section E.3 will discuss the first two effects of remanence and flux motion. Section E.3.1 presents methods for mitigating the magnetic field artifacts that have been described.

Before reading this application note, it is important to understand the basics of magnetic field control in the MPMS 3, especially the principle of the superconducting QuickSwitch, as described in section 3.4 of the *MPMS3 User Manual*. Familiarity with superconducting phenomena, especially the mixed state of type II superconductors, is also highly recommended as this note will not provide that background (see ref. <sup>i)</sup>)

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## E.3 Magnet remanence and flux motion

The effect of magnet remanence at low fields is an offset error in the reported magnetic field. This offset is dependent on the magnet history and is opposite in sign when coming from positive versus negative fields. Figure E-1 below illustrates how this offset manifests in DC magnetometer data for a sample that is magnetically reversible. If one plots the magnetic moment vs. the reported MPMS 3 magnetic field, the red curve results. Note that the hysteresis curve is “inverted” so that there is an apparently negative coercivity for the sample! On the other hand, if one were to plot the moment vs. the actual magnetic field at the sample location, the reversible blue M(H) curve results. While it is unphysical for the entire M(H) hysteresis loop to be inverted (it implies continuous heat absorption by the material as one cycles the field) it is possible to have a locally inverted region of the loop. The existence of locally inverted hysteresis loops is of interest to the research community, so it is important to discern real sample physics from instrument artifacts, as pointed out in ref. <sup>ii)</sup>.

If we define the field error as:

$$\text{field error} = (\text{real magnetic field at the sample}) - (\text{reported field})$$

and plot this quantity as a function of the field in a typical MPMS 3 magnet (see Figure E-2), we see that the absolute error decreases at higher fields, the sign of the field error depends on the direction of magnet charging, and that switching the field charging direction results in the field error traversing to the appropriate leg in a short field duration (see traversing lines at -3 and +5 tesla).

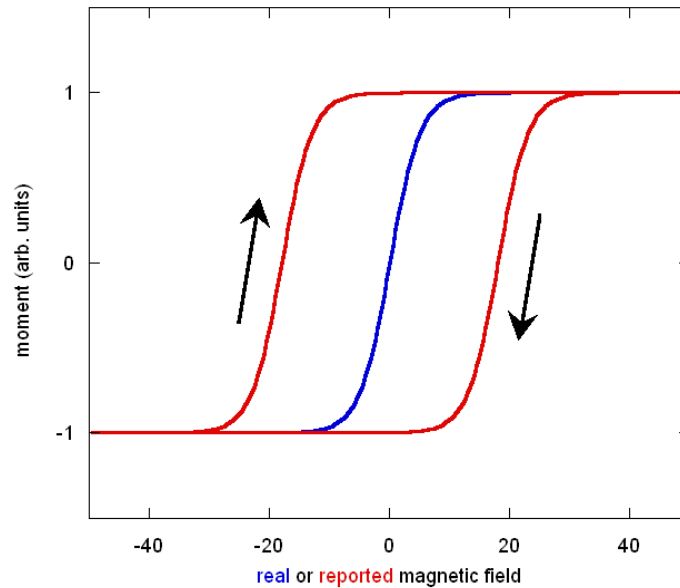


Figure E-1. A magnetically reversible material (blue curve) can appear hysteretic (red curve) due to superconducting magnet remanence. Note the inverted (clockwise) nature of the loop.

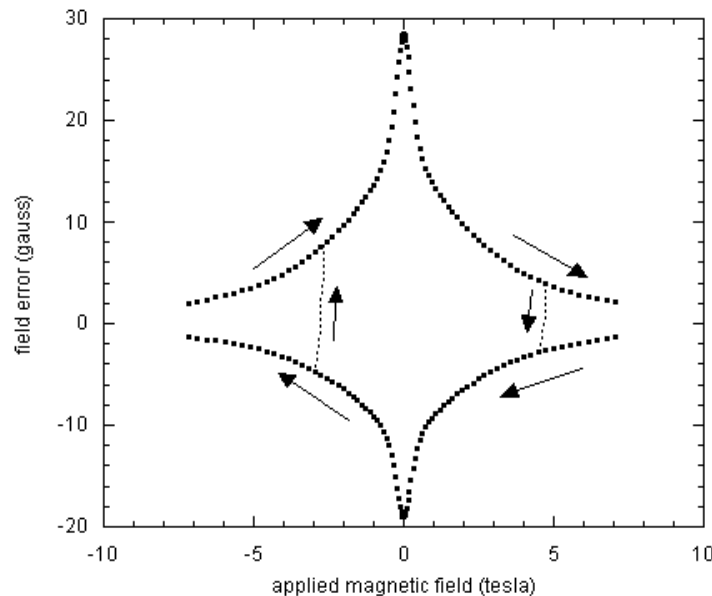


Figure E-2. Field error (defined in text) vs. the charge field in a typical MPMS 3 superconducting magnet. Note the negative sign of the field error when decreasing from positive fields. Note also that the error is generally not symmetric between up and down leg

Another important feature to notice in Figure E-2 is that the field error is negative when discharging from positive fields. That is, if setting zero field in linear charging mode from +1 tesla or above, the resulting field at the sample will be about -20 gauss in this example. So, if one wants to reach true zero field it means stopping the field discharge short at +20 Oe instead of overshooting zero field as one might intuitively think (see footnote <sup>iii</sup> for a discussion of magnetism units). Finally, note the asymmetry between the values of field error for up and down ramping directions. It has been observed that the field error magnitude near zero field can differ by up to 15 gauss (this is a property of a particular magnet) for the different charging directions.



Figure E-3 shows the typical magnitude of the field error in a 7 tesla MPMS 3 magnet. Note the log-normal plot style necessitated by the large variation in the field error as a function of applied field.

As with the other figures, this should not be used to generate a correction for data taken on a different system as the magnitude of the remanence varies from magnet to magnet. Rather, these figures should be used for their qualitative information.

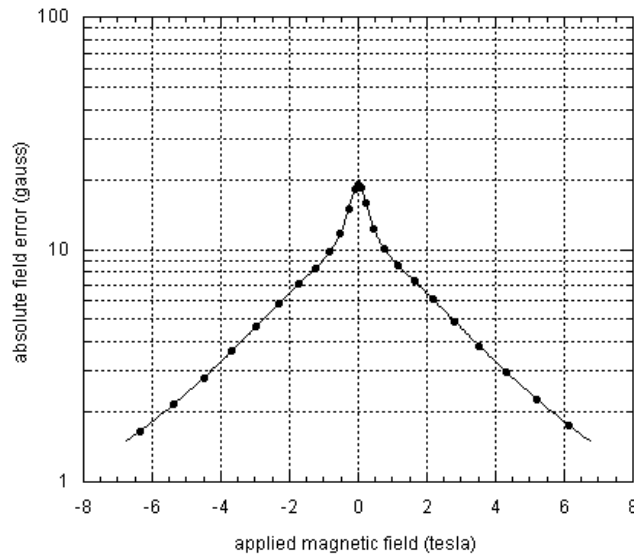


Figure E-3. Typical magnitude of average field error (averaged between up and down ramp directions) as a function of the magnet field for a MPMS 3 magnet. Note that the field error can be as much as 15 gauss different between up and down ramp directions.

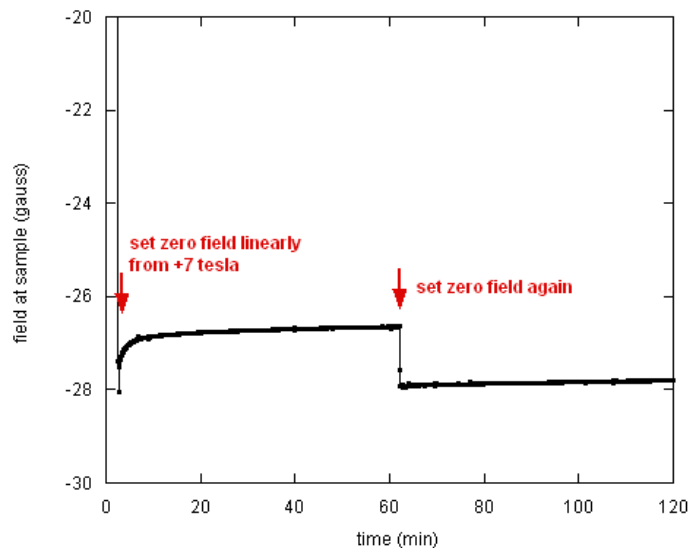


Figure E-4. The magnetic field at the sample location, as measured using a paramagnetic Dy<sub>2</sub>O<sub>3</sub> sample, slowly drifts by about 1 gauss upon setting zero field in LINEAR mode from +7 tesla. After 1 hour, the field was once again set to zero which heats the QuickSwitch and removed persistent currents. See the diagram on the next page for an explanation of this phenomenon.

The effect of magnetic flux motion on the magnetic field at the sample location is illustrated in Fig. 4. The field was set from +7 tesla to zero in linear mode and stabilized. The effects shown here are seen to be independent of the initial field providing it was larger than +1 tesla.

These unusual observations can be explained by the fact that a small amount of pinned remnant magnetic flux in the magnet escapes from the magnet windings into the bore of the magnet. In order to understand this, note that when the QuickSwitch is in the superconducting state it will sustain small DC currents without dissipation. This means that the current in the magnet can differ slightly from the current at the power supply when the QuickSwitch is in the superconducting state, a fact which should be kept in mind for the discussion below. As illustrated in the table and the accompanying figure below, a magnet is originally in its virgin state (1) in which no flux is pinned inside it. Upon heating (opening) the QuickSwitch and passing current in the magnet windings (2), a positive field is generated in the sample space at the center. After removing the current in the magnet and cooling (closing) the QuickSwitch (3), some flux remains pinned in the magnet and has the same sign as the original field because it resulted from field lines penetrating the magnet at positive fields.<sup>iv</sup> However, the stray fields from these “dipoles” are of course of the opposite sign, hence the negative initial remanent field at the sample location after discharging from positive fields. After an hour in this state, a small fraction of the pinned flux lines have escaped from the magnet windings and are in the bore of the magnet (4). Since this flux is now in free space, it must be represented by currents flowing in the magnet and these are in the direction of the original magnetic field. Since the magnet is superconducting (the QuickSwitch acts as a persistent switch at small currents like this), these currents will not dissipate. The small amount of flux escape has a large effect on the field at the sample location due to a field amplification effect: the field (seen at the sample location) when the flux has moved into the bore of the magnet is much larger than the stray field when that same amount of flux was pinned in the magnet.<sup>v</sup> Upon heating the QuickSwitch and setting zero field (5), the power supply removes the positive current in the magnet and the field that remains at the sample location is due to the stray fields from the remaining pinned vortices in the magnet. The magnetic field at the sample is nearly the same as in state (3) because the vast majority of the flux has remained pinned in the magnet.

Table E-1. QuickSwitch and Remnant Field States

<i>Step</i>	<i>magnet state</i>	<i>switch state</i>	$I_{magnet}$	$B_{sample}$
1	virgin	closed	0	0
2	charged	open	$I_2 > 0$	$B_2 > 0$
3	remanent	closed	$I_3 = 0$	$B_3 < 0$
4	remanent + 1 hour	closed	$I_4 > 0$	$B_4 > B_3$
5	remanent, reconnected	open	$I_5 = 0$	$B_5 \cong B_3$

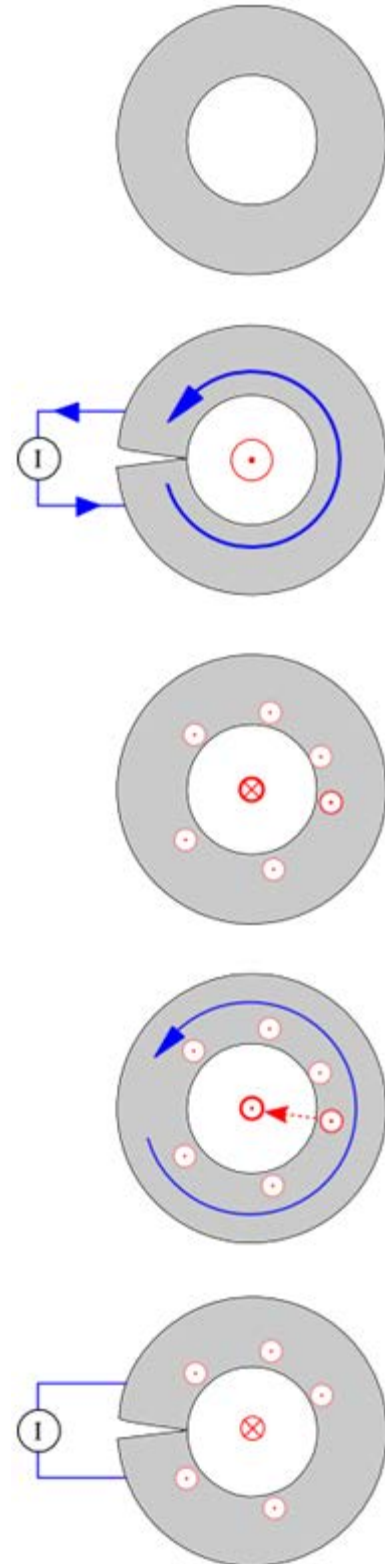


Figure E-5. Top view of magnet.

### E.3.1 Mitigating magnetic field artifacts in measurements

The magnitude of the remanence in the magnet can typically be reduced down to 1-2 gauss by setting the field to zero (from an initial field above 1 tesla) in oscillate mode. This will also attenuate the flux creep effects. Remanence can be further reduced down to  $<0.05$  gauss by using the Ultra Low Field option for the MPMS 3 which employs a fluxgate magnetometer and the modulation coil in the magnet to compensate trapped flux from the magnet.

Once a low-remanence state has been achieved in the magnet, you can make measurements at low fields ( $-100 \text{ Oe} < H < +100 \text{ Oe}$ ) without inducing a significant amount of remanence. However, a field of 1 kOe will already introduce a lot of pinned flux in the magnet.

Some measurement situations (most commonly  $M(H)$  loops from high fields through zero field) do not permit the use of low field techniques discussed above. In this case there are some guidelines we recommend in order to achieve best results:

- Determine the magnet remanence and flux creep properties of your magnet (see Figs. G-2 thru G-5) by using a pure paramagnetic sample such as the Er:YAG standard sample (4500-636) or a  $\text{Dy}_2\text{O}_3$  pellet (4041-008) that is free of any measureable amount of ferromagnetic impurities (very important – a ferromagnetic signature will lead to an underestimate of the real magnet remanence value); this option is good since in principle the  $M(H)$  slope for these materials is very nearly constant at 300 K so one can determine the absolute field error at each field – contact [apps@qdusa.com](mailto:apps@qdusa.com) for more information.
- The remanence properties of the magnet are very reproducible as long as the same field charging recipe is followed each time; thus, once the field error table is determined by using a standard sample, you can run the same field charging sequence on your sample and apply this field correction; alternately, you can modify the field set points of the measurement protocol so that the actual field is the desired one (in the example from Fig. 2, setting +20 Oe from positive high fields will produce a field at the sample of  $\sim 0$  Oe).

In conclusion, one must be cautious when using a magnet used for generating  $\sim 10^4$  gauss to also accurately generate fields on the order of 10 gauss. By understanding the basic operating principles of a superconducting magnet and by following the guidelines laid out above, it is possible to greatly reduce artifacts in the reported magnetic field.

While we have focused here on the instance of the Quantum Design MPMS 3, the effects of remanence, flux creep and persistent switch leakage currents discussed in this application note are relevant to any superconducting magnet with a superconducting switch.

<sup>i</sup> M. Tinkham, *Introduction to Superconductivity* (McGraw-Hill, 1996).

<sup>ii</sup> “Soft-magnetic materials characterized using a superconducting solenoid as magnetic source” Giovanni Mastrogioacomo, Jorg F. Löffler, and Neil R. Dilley, *Appl. Phys. Lett.* **92**, 082501 (2008), DOI:10.1063/1.2838733

<sup>iii</sup> Note on the magnetic units convention: we refer here to the magnetic field  $\mathbf{B}$  in units of gauss (where  $10^4$  gauss = 1 tesla) and it represents the real magnetic field at the sample. In contrast, the magnetic induction  $\mathbf{H}$  is expressed in oersted (Oe) and, being defined as the field due to free currents, this is the one that the user controls when setting a current at the magnet power supply. In SI units,  $\mathbf{B} = \mu_0(\mathbf{H} + \mathbf{M})$  where  $\mathbf{M}$  is the magnetization density and is defined as arising from bound currents. For more information, see the Magnetic Units Conversion Table under Technical Resources at the Quantum Design website [www.qdusa.com](http://www.qdusa.com).

<sup>iv</sup> For viewing clarity in the figure, the magnetic field lines in the superconductor do not show the supercurrent vortices that generate the magnetic field.

<sup>v</sup> Consider the persistent magnet as a superconducting ring of inner dimension of  $R$  and the pinned flux line in the superconductor with a radius  $r$ , where  $R \gg r$ . Referring to the states in Table 1, we must compare the magnetic field at the sample location (taken as the origin) between the initial remanent state (3) and when the vortex moves out of the ring into the middle (4).

$$\text{We know that flux is conserved: } \Phi = B_3(R) \cdot r^2 = B_4(0) \cdot R^2$$

That is, the flux enclosed on vortex at position  $R$  (at the inner bore of the magnet) is compared with field in the bore of the magnet after the vortex moves out.

The equation above approximates the magnetic field  $B$  as constant over the area of the loops in both cases. While this is not strictly true, the error is small compared with the amplification effect we are observing and hence is neglected.

$$\text{magnetic moment of vortex: } m = \pi r^2 \cdot I$$

where  $I$  is the current flowing around perimeter.

$$\text{magnetic field at center of the vortex (a current ring): } B_3(R) = \frac{\mu_0 \cdot I}{2r}$$

$$\text{put this value of } I \text{ into above expression for moment: } m = \frac{2\pi}{\mu_0} B_3(R) r^3$$

$$\text{magnetic field at sample location due to pinned flux “dipole”: } B_3(0) = -\frac{\mu_0}{4\pi} \cdot \frac{m}{r^3}$$

$$\text{insert expression for } m \text{ from above: } B_3(0) = -\frac{B_3(R)}{2} \cdot \left(\frac{r}{R}\right)^3$$

Use flux conservation equation to compute ratio of field seen at sample in state 4 vs. state 3:

$$\frac{B_4(0)}{B_3(0)} = -2 \frac{R}{r}$$

Since  $R \sim 2.5\text{cm}$  and  $r \sim 10^{-3}\text{ cm}$ , this amplification is over x1000.

This is why a tiny amount of flux creep out of magnet will have a large effect on the field seen at the sample location.






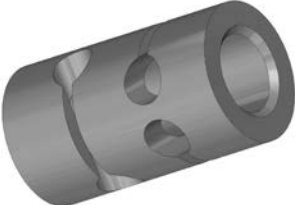
# A P P E N D I X F

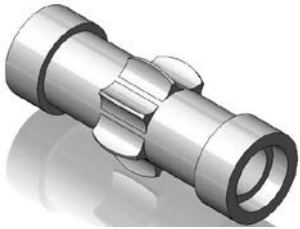
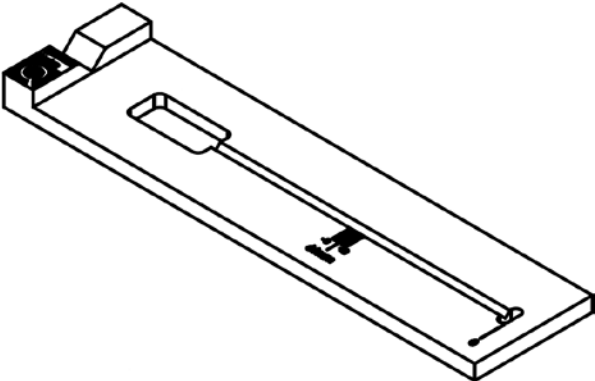


## Ordering Replacement Parts

### F.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 3. Contact your local Quantum Design representative to order parts.

Table F-1. Replacement 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

	<p>Sample rod bearing</p>	<p>4500-603</p>
	<p>sample mounting station</p>	<p>4500-626</p>
	<p>ultratorr fitting (tube fitting) for helium transfers</p>	<p>YSS-6-UT-A-8BT</p>
	<p>nitrogen fill port plug</p>	<p>4504-032</p>
<p>pump oil</p>	<p>HH-110-25-012</p>	
<p>activated alumina for oil mist filter on rotary vane pump</p>	<p>H026-00-050</p>	
<p><b>CONSULT A QUALIFIED QUANTUM DESIGN SERVICE REPRESENTATIVE BEFORE REPLACING ANY O-RINGS OR SEALS.</b></p>		
<p>teflon gas cap seal used on helium inlet plug</p>	<p>4504-002</p>	
<p>o-ring for cap seal used on helium inlet plug</p>	<p>VON2-014</p>	
<p>o-ring for nitrogen fill ports</p>	<p>VON2-012</p>	
<p>o-ring for transfer line bayonet</p>	<p>VON2-014</p>	
<p>o-ring for top of sample space manifold</p>	<p>VON2-019</p>	

o-ring for motor cap	VON2-022
<b>CONSULT A QUALIFIED QUANTUM DESIGN SERVICE REPRESENTATIVE BEFORE REPLACING ANY FUSES</b>	
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



