

Quantum Design



VersaLabTM
free

User's Manual

Part Number 1300-001, B2

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Contents and Conventions

P.1 Overview

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.
- Section P.5 describes the safety guidelines and regulatory information in the manual.

P.2 Scope of the Manual

This manual discusses the Quantum Design VersaLab instrument. It contains information about basic functionality, describes the system hardware, and the control software.

Most VersaLab systems include measurement options that you use for performing sample measurements. This manual discusses how measurement options interface with VersaLab, but does not cover any details about specific options. Be sure to read separate measurement option user manuals.

All VersaLab systems include a cryocooler system, which consists of a cold head and a compressor manufactured by Sumitomo Heavy Industries. Please read the supplied manuals about these components.

P.3 Contents of the Manual

- Chapter 1 provides an overview of the operation of the VersaLab.
- Chapter 2 provides an overview of the hardware.
- Chapter 4 describes the VersaLab System Operation.
- Appendix A contains maintenance interval information as well as fuse and O-rings specifications.

- Chapter 3 describes the user's interface and software.
- Alignment Appendix B provides a description of diagnostic software operations.
- Appendix C describes system's voltage, frequency and power requirements, pin out and interconnect diagrams.

P.4 Conventions in the Manual

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

CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.



WARNING!

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



ELECTRIC SHOCK!

This symbol signals electrical hazards that could result in bodily harm, or loss of life. Used at all accessible 200-230 V and 380-408 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).



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 users 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.



This symbol signals information on **fusing**.


P.5 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 VersaLab 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.


This product is NOT user serviceable except for the operations that are described as performable by the user in Appendix A.

Observe the following safety guidelines when you use your system:


	WARNING!
	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.5.1 Inspection for Damage

The VersaLab 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.

	WARNING!
	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.5.2 Pressurized Gas

	WARNING!
	The helium compressors used with the VersaLab are charged with helium gas at high pressure. Typical pressures found in the system can range from 0.1-2.5 MPa. This requires the operator to follow appropriate safety protocols.

Always wear protective clothing, including eye protection, gloves and covered shoes when you work with pressurized gas. The compressor is equipped with low loss fittings, so attaching and removing helium hoses does not require a total venting of the system.

- Quantum Design recommends installing oxygen sensors where the VersaLab is located and with all other cryogenic systems.
- Work with high gas pressure lines only in well-ventilated areas. In the event of a gross helium gas leak from the gas lines, vent the room immediately and evacuate all personnel. In a poorly ventilated area, helium can displace the air, leading to asphyxiation. Because helium rises, well-ventilated rooms with high ceilings are generally the safest.
- Arrange the compressor lines, power and connectivity cable, and helium supply lines in a neat manner to avoid tripping hazards.
- Do not pinch the high pressure lines from the helium compressor or bend them with a radius tighter than 60 cm (24 inches). These lines are at a pressure of about 15 bar (300 psi). A puncture in the helium lines might cause an explosion and rapid escape of high pressure gas.
- Wear hearing protection whenever performing a cold head vent to avoid hearing loss.

P.5.3 Electricity



WARNING!

High voltage is supplied to the compressor making a shock hazard possible if inadequate safety procedures are followed.


- In case of emergency, switch the power off at the back of the helium compressor or unplug the main power cords from the wall power outlets.
- 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. To prevent electrical shock, disconnect the compressor from the power before you install it, adjust it, or service it.
- Keep electrical cords in good working conditions, 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 Section A.6.
- In general, keep liquids away from the VersaLab and helium compressor unit.
- Keep the VersaLab and helium compressor away from radiators and heat sources. Provide adequate ventilation to allow for air cooling around the compressor.

- Remove the Power and Connectivity cable from the back of the VersaLab before connecting or disconnecting the cold head cable. Failing to observe this precaution may result in electrical shock.
- If the compressor is used in a way not specified by Quantum Design, then the protection provided by the equipment may be impaired.
- Be sure to follow circuit breaker specifications outlined in the helium compressor manual.

P.5.3.1 TURNING OFF THE HELIUM COMPRESSOR

- Make sure the helium compressor is turned off before connecting or disconnecting the cold head cable. Failing to observe this precaution may result in electrical shock.
- Set helium Compressor to “OFF” state by depressing once the “RUN” button on the front control panel. The front panel will then display: “OFF by REMOTE CTRL”. Note that it might take a few minutes for the Cold Head to stop. Once the Cold Head is completely stopped proceed to the next step.
- Power down the indoor compressor by toggling the switch in the back of the indoor compressor control unit. This will shut down any 200-240 V power.
- Disconnect the 200-240 V power by turning off the Low Voltage breaker.
- Unplug the compressor power cords from the power source if needed

P.5.4 Moving the VersaLab

	WARNING!
Never operate the VersaLab on uneven surfaces or on inclines greater than a 1:12 ratio.	

VersaLab may be moved in one of two ways:

- One person may roll VersaLab to the desired location using the wheels on the back of the assembly. To do this, place one foot on the wheel axle and pull back on the round stainless steel handle at the rear of VersaLab as shown in Figure P-1. Then simply roll the instrument to the desired location. Take care not to suddenly lower the instrument, as doing so could cause damage.
- Two people may simply lift VersaLab and carry it. To do this, two people stand, one on either side of VersaLab, and pick up the instrument by the rectangular cross-section stainless steel bar. Carry it to the desired location and carefully set it down.

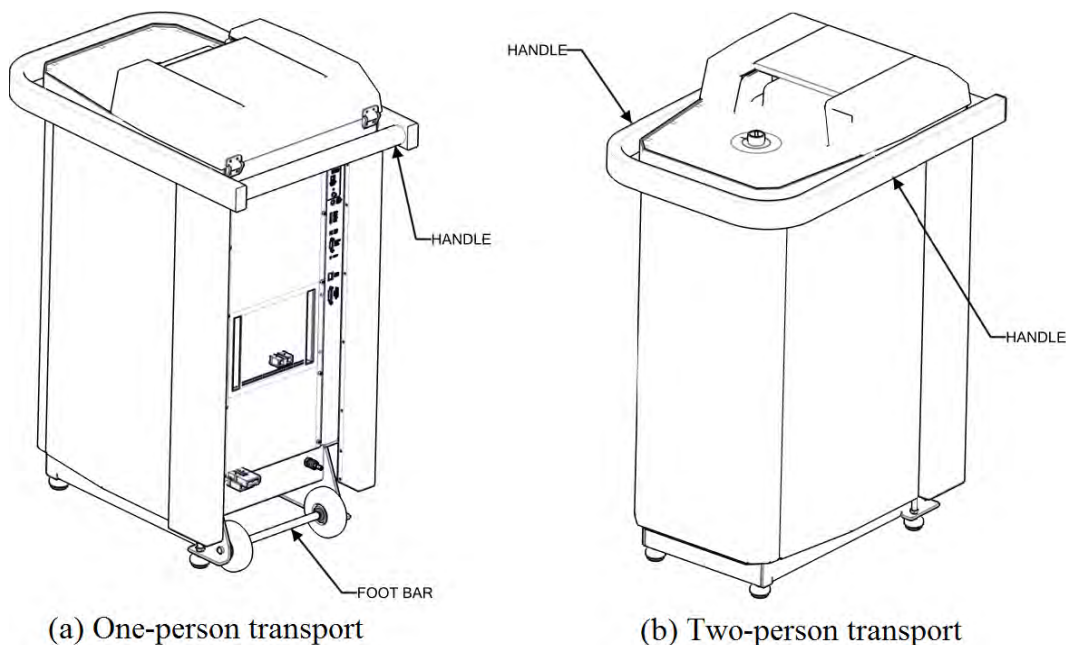



Figure P-1. How to move the VersaLab with one person (a), or two people (b).

	WARNING! Be careful when moving VersaLab. Do not drop the instrument, because doing so could cause damage. Do not attempt to lift the instrument by yourself, because doing so could cause injury. Take care not to damage the high-pressure gas lines when moving the instrument.
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P.6 Disposal Information

The VersaLab 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 VersaLab 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.6.1 RoHs Statement





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

P.6.2 WEEE Statement

WEEE Statement: The VersaLab 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 the time of decommissioning for proper handling before recycling/disposal. Contact Quantum Design for updated procedures/recommendations before disposal.

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

<i>Component Description / Location</i>	<i>Identifying Photograph/Diagram</i>
VersaLab Controller (QD P/N.: 7302-251)	
Cryogenic Cooling System: Sumitomo cold head, RDK 101 with integrated motor (QD P/N.: 4302-040-01).	
VersaLab Backplane (Qd P/N.: 3305-020)	
H-Plate VersaLab (QD P/N.:4372-038)	

<p>Display Panel Assembly (QD P/N.: 4601-061-01)</p>	
<p>Compressor Controller Assembly (QD P/N: 4601-130-01)</p>	
<p>Helium Scroll Capsule (QD P/N: HM504)</p>	
<p>Electronics Bay Assembly (QD P/N: 4601-160)</p>	

Getting Started

1.1 Overview

This chapter provides a brief introduction to the VersaLab and describes how to perform some common tasks. Be sure to read the rest of the manual to become familiar with the details of the system.

1.2 Introduction

The Quantum Design VersaLab is an instrument designed to measure a variety of a sample's physical properties while controlling the conditions experienced by the sample: temperature (400 to 50 K), magnetic field (up to 3 T), and atmosphere (atmospheric pressure to high vacuum). VersaLab is a cryogen-free instrument, so no costly liquid cryogenes are required to operate the instrument. Instead, VersaLab uses a cryocooler to achieve cryogenic temperatures. VersaLab is a highly automated instrument, so it can perform unattended measurements after the user has installed a sample and configured the measurement.

VersaLab is a very flexible instrument, able to perform a wide variety of measurements, including magnetic, electrical transport, heat capacity, and thermal transport. VersaLab achieves this flexibility through the use of a standardized cryogenic electrical and thermal interface (the puck interface), modular measurement electronics (CAN modules), and modular software design (MultiVu plus measurement option software).

VersaLab is similar to another Quantum Design instrument, the Physical Property Measurement System (PPMS). VersaLab and the PPMS share the sample puck interface, sample Lemo connector wiring, and measurement option hardware. VersaLab and the PPMS use similar versions of the MultiVu software for instrument control and automation. The software for measurement options is also quite similar in the two instruments. If you are a PPMS user, much of what you know about operating the PPMS will help you to operate VersaLab. However, there are important differences between the two instruments, so you should read this manual.

The rest of this chapter describes some common tasks performed when using VersaLab. After reading this chapter, be sure to read the rest of the manual in order to learn more about the hardware (Chapter 2), software (Chapter 3), and system operation (Chapter 4). You may also want to consult the appendices to learn about maintenance (Appendix A), diagnostic software (Appendix B), and interconnect and pin-out diagrams (Appendix C).

1.3 Common Tasks

The most common tasks you perform when using VersaLab are those required to carry out a sample measurement: mounting the sample, inserting a sample puck into VersaLab, and setting up a measurement option. The sections below describe these procedures.

1.3.1 Sample Mounting

Sample mounting techniques vary widely depending on the type of measurement you perform. Consult the appropriate measurement option manual for information about sample mounting methods. This section describes some general issues to consider when mounting samples. Also, if you are not using a measurement option supplied by Quantum Design, consult this section for mounting information.

Several broad considerations affect the choice of sample mounting technique in VersaLab. Consider the following list before choosing a technique.

- **Thermal contact.** Good thermal contact is required in order for the sample chamber thermometer (or option thermometer) to accurately report the sample temperature. Securely mounting the sample to a puck is one way to achieve good thermal contact. Two common mounting techniques are to use grease or varnish. Be sure that any grease you use does not melt in the temperature range of interest.
- **Electrical contact.** For many measurements you must make good electrical contact to your sample. Exactly how you do this depends on the details of your sample. Example techniques include solder, welding, and metal-filled paints.
- **Ground isolation.** In general, the sample must be isolated from ground if you are performing electrical measurements. The puck is grounded, so you must isolate your sample from the puck. One way to electrically isolate a sample, but still achieve good thermal contact, is to place cigarette paper soaked in varnish between the puck surface and sample. Samples already mounted to an insulator, such as a thin film on an insulating substrate, require no additional isolation.
- **Magnetic background.** If you are performing magnet measurements, consider the magnetic background of your mounting hardware. Consider measuring an empty sample holder to determine the size of the background.
- **Differential contraction.** As temperature changes, materials contract at different rates. This may cause leads to pop off the sample, the sample to pop off the puck, or epoxy joints to fail. Consider using thermally matched materials to avoid this problem. Any material, such as epoxy, that is not well thermally matched, should be kept as thin as possible.
- **Secure mounting.** Your sample should be mounted securely. If it falls off during measurement, it will be very difficult to retrieve from the sample chamber.
- **Clearance.** Your sample mounting technique must allow clearance for insertion into the instrument. For a sample mounted to a puck, everything must fit inside the puck insertion tool, and the groove on the outer rim of the puck should be clear for gripping by the insertion tool. Some measurement options, such as the Vibrating Sample Magnetometer, have sample clearances set by option hardware. See the relevant measurement option for details.

After mounting your sample, it is useful to check electrical connections before inserting the puck into VersaLab. Use the puck wiring test station to test your connections (Figure 1.1). Once the puck is inserted into the test station, you may test all sample connections using the banana jacks.

Be sure to also check for isolation between ground (the puck) and your sample leads. Templates are supplied with measurement options to aid you in identifying the leads. A 14-pin Lemo connector, identical to the VersaLab sample wiring Lemo connector, allows connection to measurement option hardware for testing measurement options without inserting a puck into VersaLab. The Lemo connector, puck connector, and banana jacks are all wired in parallel.

The test station can also be used to test pucks that are wired by Quantum Design, such as Heat Capacity and VSM coil set pucks.

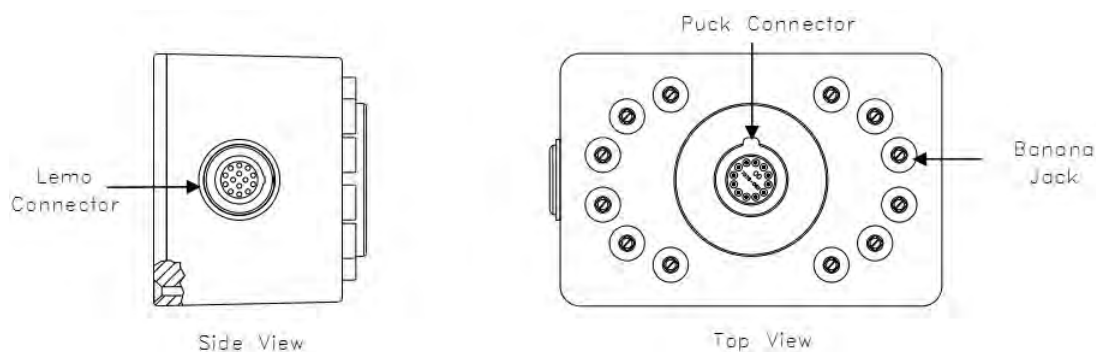


Figure 1-1. Puck-wiring Test Station

For user-designed measurements, you may wish to modify the puck to accommodate your hardware. To do so, first unscrew the single screw in the center of the bottom of the puck and remove the PC board assembly. The metal chuck can then be machined to add the desired features or mounting holes. Be sure not to damage the delicate fingers or the groove used by the insertion tool. When reinstalling the PC board assembly, be sure to tighten the screw securely so that it does not come loose upon thermal cycling in the instrument. Otherwise, you might lose the PC board assembly in the bottom of the sample chamber.

1.3.2 Sample Puck Installation and Removal

1.3.2.1 INSTALLING A SAMPLE PUCK

A puck that you are installing in the sample chamber may have your sample mounted on it, or it may have measurement hardware on it, e.g. VSM coil set. Either way, use the following procedure to install a puck. For a puck with a sample on it, you should use the measurement option sample installation wizard to walk you through this process, because the wizard also guides you through other important tasks such as setting up a data file. The measurement option will not give you detailed instructions on use of the puck insertion tool, so be sure to read these instructions first.

1. Disengage the sample insertion tool by flipping up the black switch located on top of the tool or by fully depressing the switch. Refer to Figure 1-2.

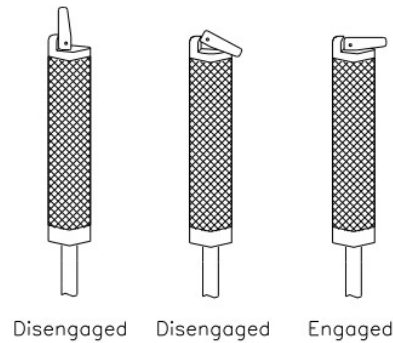



Figure 1-2. Operation of Puck Insertion Tool

2. Insert the puck, with the sample facing upward, inside the hollow cylinder that is at the bottom of the sample insertion tool. The sample will be inside the cylinder and the connectors and solder pads will be outside the cylinder. Refer to Figure 1-3.
3. Rotate the puck to verify that it is properly seated inside the hollow cylinder. The puck should rotate smoothly.
4. Engage the sample insertion tool by flipping down the black switch located on top of the tool or by releasing the switch, if it is fully depressed, so that the switch lies flat across the tool's handle. Refer to Figure 1-3. The tool should now grip the outer rim of the puck.
5. Verify that the puck remains properly seated in the hollow cylinder. The puck must be level, and should not rotate easily. Gently pull on the puck to verify that the tool holds it securely.



WARNING!

An unlevel puck can get stuck in the sample chamber and can damage the pins at the bottom of the chamber. Before inserting the puck into the chamber, you must verify that the puck is level within the cylinder of the sample insertion tool.

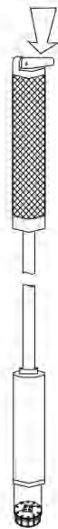


Figure 1-3. Puck Insertion Tool Connecting to Puck

6. Verify that the sample chamber is at or above 298 K. The temperature must be at least 298 K to prevent condensation of water or cryopumping of air into the chamber. If the temperature is below 298 K, set it to 298 K and wait for the chamber to warm up. Use MultiVu to set the temperature: click on the temperature display in the status bar. In the temperature dialog, enter 298 K, 20 K/min, and select Fast Settle mode. Press the Set button. An installation wizard in measurement option software may set the temperature for you.



WARNING!

Do not open the sample chamber if it is below room temperature. Doing so may condense water and air in the sample chamber. Such contaminants may form a plug in the chamber resulting in dangerous and damaging over pressure condition on warming.

7. Verify that the magnetic field is less than 1 T (10,000 Oe). If the magnetic field is greater than 1 T, set zero field and wait for the field to stabilize before continuing. Use MultiVu to set the field: click on the field display in the status bar. In the field dialog, set 0 Oe, 300 Oe/sec, and select Linear approach. Press the set key.

CAUTION!

If you place the sample insertion tool into the sample chamber when the magnet is at high field, the insertion tool will be strongly pulled into the sample chamber. The force may overwhelm you and cause damage to the instrument.

8. Vent the sample chamber with clean, dry gas. Use MultiVu: click on the chamber atmosphere display in the status bar (this display shows the pressure and chamber state.) Press the “Vent Cont.” button in order to vent the chamber continuously. Do not leave the chamber in this state for longer than necessary, because doing so wastes vent gas from the bottle you supply. An installation wizard may vent the chamber for you.
9. Open the hinge clamp and remove the KF blank flange (and attached baffle set, if present) from the sample chamber access port. If the blank flange is difficult to move due to low pressure in the chamber, do not force it. Allow the pressure within the chamber to match the external pressure before you open the sample chamber to atmosphere.
10. Remove the O-ring from the sample chamber access port.
11. Gently lower the sample insertion tool into the sample chamber puck-end first until the sample puck touches the puck connector at the bottom of the chamber. Do not force the puck down farther once it touches the connector.
12. Rotate the sample insertion tool slowly until the key on the puck drops into the indexing notch. When the puck drops into the notch, you feel the puck lock into position.
13. Gently push down on the sample insertion tool in order to engage the puck interface and to make solid electrical contact between the interface and the puck.



WARNING!

Do not use excessive force when engaging the puck interface. The need for excessive force may indicate a problem, such as a misaligned puck. If you push hard you may bend the pins at the bottom of the sample chamber.

14. Disengage the sample insertion tool, and then raise the tool several centimeters. Be sure there is no resistance when you raise the insertion tool. Resistance indicates the puck may have gotten caught in the tool as you began lifting it out, and you may thus need to remove the puck and try inserting it again.
15. Remove the sample insertion tool from the sample chamber.
16. Check that the sample chamber access port KF flange, blanking flange, and O-ring are clean. Place the O-ring over the port, and replace the blank flange (with attached baffle set).
17. Place the flange clamp in position around the top of the sample chamber access port, and then latch the clamp.
18. Purge and seal the sample chamber using MultiVu: click on the chamber atmosphere display in the status bar. Press the Purge/Seal button in order to purge and seal the sample chamber. An installation wizard may do this for you.

The system is ready to begin an experiment. You should refer to the appropriate measurement option manual to determine what to do next. Once you perform a measurement and verify instrument operation, you may want to write a sequence to automate the measurement. See Section 3.4 for a discussion of sequences.

1.3.2.2 REMOVING A SAMPLE PUCK

The procedure for removing the puck from the sample chamber is essentially the reverse of the installation procedure. Complete the following steps to remove the puck:


1. Verify that the sample chamber is at or above 298 K. The temperature must be at least 298 K to prevent condensation of water or cryopumping of air into the chamber. If the temperature is below 298 K, set it to 298 K and wait for the chamber to warm up. An installation wizard in measurement option software will set the temperature for you.



WARNING!

Do not open the sample chamber if it is below room temperature. Doing so may condense water and air in the sample chamber. Such contaminants may form a plug in the chamber resulting in a dangerous over pressure condition on warming.

2. Verify that the magnetic field is less than 1 T (10,000 Oe). If the magnetic field is greater than 1 T, set zero field and wait for the field to stabilize before continuing.

	WARNING!
	If you place the sample insertion tool into the sample chamber when the magnet is at high field, the insertion tool will be strongly pulled into the sample chamber. The force may overwhelm you and cause damage to the instrument.

3. Vent the sample chamber with clean, dry gas. An installation wizard may do this for you.
4. Open the hinge clamp and remove the KF blank flange (and attached baffle set, if present) from the sample chamber access port. If the blank flange is difficult to move due to low pressure in the chamber, do not force it. Allow the pressure within the chamber to match the external pressure before you open the sample chamber to atmosphere.
5. Remove the O-ring from the sample chamber access port.
6. Disengage the sample insertion tool by flipping up the black switch located on top of the tool or by fully depressing the switch. Refer to Figure 1-2.
7. Gently lower the sample insertion tool into the sample chamber cylinder-end first until the tool touches the bottom of the chamber.
8. Engage the sample insertion tool by flipping down the black switch located on top of the tool or by releasing the switch, if it is fully depressed, so that the switch lies flat across the tool's handle. Refer to Figure 1-3.
9. Gently raise the insertion tool out of the sample chamber. You should feel some initial resistance as you pull the puck out of its seat.
10. Verify that the sample puck is in the insertion tool. If it is not, return to step 6. If it is, disengage the lever and let the puck fall safely into your hand. *Do not drop the puck.*

You may insert another puck, install measurement option hardware into the sample chamber, or close the sample chamber at this point. To close the sample chamber, you (1) place the O-ring and KF blank flange over the sample chamber access port, (2) place the flange clamp in position around the top of the chamber access port, (3) latch the clamp, and then (4) purge and seal the sample chamber.

1.3.2.3 OPTION START UP

Most measurements that you perform in VersaLab require the use of a measurement option, such as Vibrating Sample Magnetometer or Heat Capacity. Whenever you first start using an option, or when you change from using one option to another, follow the steps outlined below. Consult the relevant measurement option manual for details about the measurement option you are using.

1. If necessary, uninstall any hardware from a measurement option previously in use.
 - a. You always uninstall all option hardware from the sample chamber, including pucks, specialized baffle sets, and hardware attached to the chamber KF flange (e.g., VSM linear motor). Be sure the chamber is at room temperature, the magnetic field is zero, and the chamber is venting continuously before attempting to remove any option hardware.
 - b. It is not necessary to uninstall CAN modules unless you need extra space in the module bay.
2. Install hardware for the measurement option you are ready to use.
 - a. For some options, you install pucks, specialized baffle sets, and hardware attached to the sample chamber. See the option manual for details. In general, you **do not** install samples, including pucks with samples on them, at this time.

- b. If necessary, install any CAN module(s) needed for the option into the module bay. The module(s) may already be installed from previous use of the option. See Section 2.6.7 for details on insertion of CAN modules.
3. Activate the option software. In MultiVu, select the menu item “Utilities > Activate Option...” You will be presented with a dialog showing available options. Select the desired option and press the “Activate -->>” button. The option software will start within the MultiVu window, and will initialize the option hardware.
4. Install your sample. Use the measurement option installation wizard, because it guides you through all the necessary steps, including setting up data files and entering sample properties.
5. Perform an immediate mode measurement using the measurement option control panel in order to verify that everything is working correctly.
6. Write a sequence to automate the measurement. Execute the sequence, and allow the instrument to acquire data. See Section 3.5 for information about writing and executing sequences.
7. When the measurement sequence is complete, remove your sample using the option’s wizard. Then either install a new sample to measure, or go back to step 1 to use a different measurement option.

C H A P T E R 2

Hardware

2.1 Overview

This chapter describes the VersaLab hardware components and their functions. You should have a basic understanding of the theory of operation of the system after reading this chapter. Although this chapter will help you understand how to operate the instrument, user operation is not described in this chapter. See Chapter 1: Getting Started, and Chapter 4: System Operation for operating instructions.

The VersaLab instrument is entirely contained within a single enclosure that includes the cryostat and electronics cabinet. The compressor and control computer are separate from this enclosure.

2.1.1 Cryostat

The cryostat assembly is contained in the front half of the VersaLab enclosure. Figure 2.1 shows a cutaway view of the cryostat.

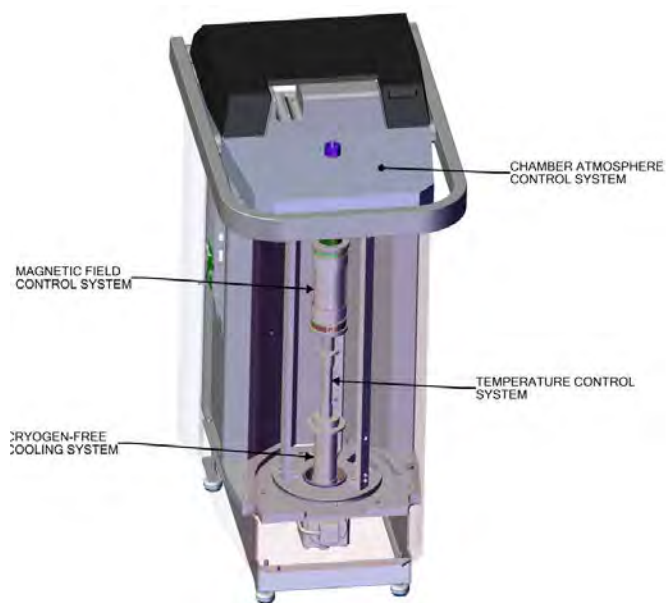


Figure 2 - 1. Main Cryostat Systems

The cryostat consists of the following systems, described in the sections below:

- Cryogen-Free Cooling System. This system allows VersaLab to achieve cryogenic temperatures without the need for liquid cryogens, and is described in Section 2.2.
- Temperature Control System. This system controls and measures the temperature of the sample, and is described in Section 2.3.
- Magnetic Field Control System. This system controls the magnetic field at the sample, and is described in Section 2.4.
- Chamber Atmosphere Control System. This system controls the gas atmosphere at the sample, and is described in Section 2.5.

2.1.2 Electronics Cabinet

The electronics cabinet is contained in the rear half of the VersaLab enclosure. This cabinet contains all of the hardware needed to control the cryostat systems. See the cryostat sections for descriptions of the electronic components used by each cryostat system.

The electronics cabinet contains the following components:

- Controller Board, which is involved in the control of all VersaLab systems.
- Magnet Controller, which supplies current to the superconducting solenoid.
- Module Bay, which houses measurement option CAN Modules.
- CANOpen-USB dongle, which allows the computer to communicate with the CAN bus via USB.
- Power supply, which supplies +24 V and -24 V to all electronics, as well as the current to the magnet controller.

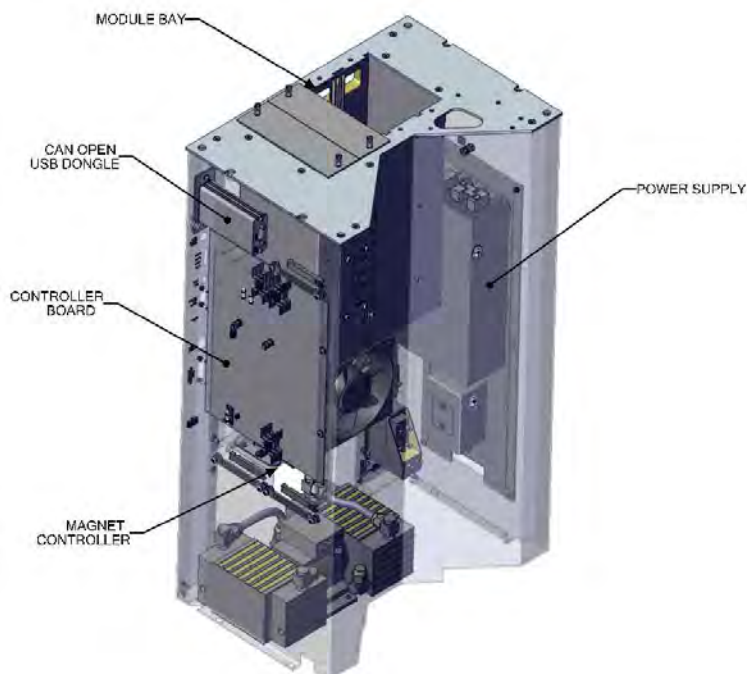


Figure 2 - 2. Main Components of the Electronics Cabinet

2.1.3 SROMs

Serial Read-Only Memory chips, or SROMs, are present in several places in VersaLab. The SROMs are used to store calibration information about the components they are attached to. The calibration information is read at runtime, reducing the need for calibration files on the computer.

There are 3 SROMs always present in VersaLab:

- Main SROM. This SROM is on the controller board, and is used to store general information about the configuration of VersaLab.
- Cryostat SROM. This SROM is on a PC board attached to the sample chamber, and is used to store cryostat calibration information, including thermometer tables and magnet calibration.
- Magnet Controller SROM. This SROM is in the Hybrid Feedback Enclosure on the Magnet Controller, and is used to store Magnet Controller calibration information.

In addition, measurement options have SROMs to store calibration information for measurement hardware.

2.1.4 Control Computer

A standard personal computer is used to control VersaLab. The computer connects to VersaLab via USB through the CANOpen-USB dongle.

2.2 Cryogen-Free Cooling System

The core difference between VersaLab and traditional cryostats is the cryogen-free cooling system. This system allows VersaLab to perform temperature dependent measurements in a magnetic field without the use of any liquid cryogens. The components of the cryogen-free cooling system are described below.

2.2.1 Cryocooler

VersaLab is cooled using a two-stage helium cryocooler comprised of a compressor, cold head, and high pressure gas connecting lines. The compressor provides helium gas at approximately 2 MPa to the cold head through high pressure flexible gas lines. Cooling is accomplished when this high pressure helium gas is expanded in the cold head. The expanded gas is then recompressed in the compressor. A displacer in the cold head, driven by a motor at room temperature, provides regenerative exchange to the helium gas. A cable connects this motor to a drive circuit located on the compressor.

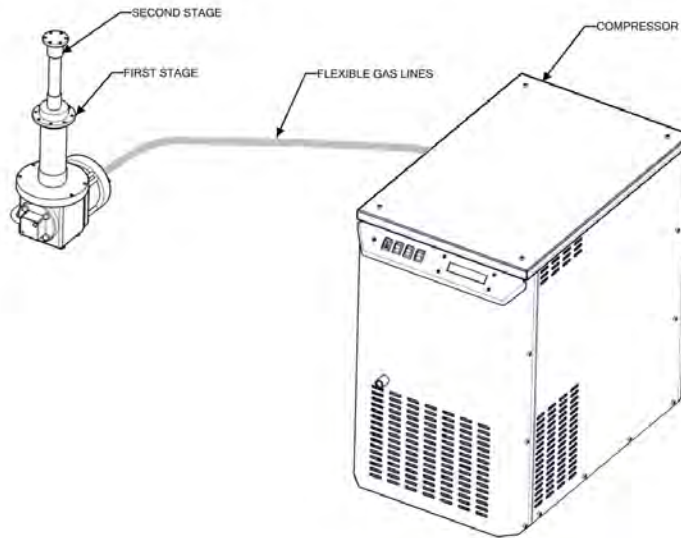


Figure 2 - 3. VersaLab Cold head and Compressor

The cold head provides cooling in two stages. The first stage provides approximately 5 W of cooling power at 60 K, and the second stage provides approximately 0.1 W at 4.2 K. The first stage of the cooler is used for many cooling tasks, including the sample chamber, thermal shields, and capturing heat leaks from components connected at room temperature. The second stage is used primarily to cool the superconducting magnet and the charcoal pot of the thermal switch. The sections below detail how each system is cooled by the cryocooler.

See Section 4.2.1 for instructions on how to set up the compressor and connect the high-pressure helium lines. See Appendix A.4 for cryocooler maintenance information. For a detailed description of the cryocooler, consult the manufacturer's manual that was included with your system.

2.2.1.1 CAUTION AGAINST HAZARD

WARNING!

In order to avoid damage and bodily injury due to the high pressures and low temperatures present in the cryocooler, follow these precautions



- Take care to not damage the high-pressure helium lines, as doing so could cause sudden release of gas.
- Do not bend the high-pressure gas lines with a radius smaller than 30 cm (12 inches). Doing so could cause damage to the lines and sudden release of gas.
- Do not connect or disconnect the high-pressure helium lines without consulting Quantum Design.
- Never disconnect the high-pressure helium lines while the system is cold. Doing so causes extremely high pressure in the cold head, resulting in loss of gas through a check valve and possible damage to the cold head.
- Do not attempt to perform any maintenance on the compressor or cold head without consulting Quantum Design.

2.2.2 Thermal Isolation

The cold components of the VersaLab cryostat must be thermally isolated from each other and room temperature. Vacuum and thermal shields provide this isolation.

2.2.3 Vacuum and Charcoal Sorbs

The VersaLab thermal isolation vacuum is provided by charcoal sorbs connected to the first and second stage of the cryocooler. Before cooling the system down, the diaphragm pump evacuates the cryostat case to < 2 Torr. During cool down, the charcoal adsorbs the remaining gas in order to provide high vacuum isolation. Note that very small quantities of helium present during a cool down can cause a high enough heat leak to prevent a proper cool down. You avoid the presence of helium by utilizing the cool down wizard described in Section 4.3.

Permeation through the various O-ring seals in VersaLab could ruin the isolation vacuum. However, the charcoal sorbs (and other cold surfaces) adsorb permeated gases, maintaining the vacuum. When the system is warmed up, these gases desorb. Therefore, the case vacuum needs to be pumped down as described in Section 4.3 before every cool down.

2.2.4 Thermal Shields

Even with a perfect isolation vacuum, thermal radiation can result in large heat leaks to the cold cryostat components. Thermal shielding reduces this heat leak. Most cold components reside inside a shield cooled by the cryocooler first stage. Superinsulation reduces the radiation heat load on this shield. A shield cooled by the cryocooler first stage between the magnet and sample chamber allows the magnet temperature to be < 4 K while the sample chamber is at 400 K.

2.2.5 Diagnostic Thermometers


VersaLab is equipped with diagnostic thermometers in order to help determine whether or not the cryogen-free cooling system is operating correctly. Diagnostic thermometers are present on the cryocooler first and second stages, the magnet, and the thermal switch charcoal pot. These temperatures are available in Log Data as described in Appendix B.2. Typical values for these diagnostic temperatures are given in Appendix A.4.1.

2.3 Temperature Control System

There are four main components in the VersaLab temperature control system, described briefly here. These components, other than the cryocooler, are described in more detail in the sections below:

- The sample chamber, which contains the sample puck, thermometers for measuring the temperature at the puck, and heaters to provide warming.
- The thermal switch, which provides a variable thermal link between the sample chamber and cryocooler first stage.
- The cryocooler. The first stage of the cryocooler is connected to the sample chamber via the thermal switch. The second stage of the cryocooler cools the charcoal pot on the thermal switch.
- VersaLab control electronics, which measure thermometers and apply appropriate heater powers to achieve temperature control.

WARNING!



Note that unlike the PPMS sample chamber, the VersaLab sample chamber is not removable from the top of the instrument. The VersaLab sample chamber is connected via flexible links to components inside the cryostat. Do not attempt to remove the sample chamber from VersaLab, because doing so can damage the instrument.

2.3.1 Thermal Switch and Thermal Circuit

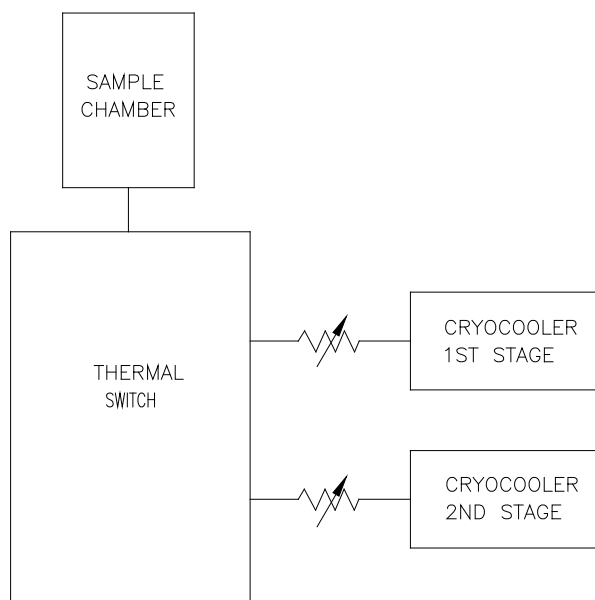


Figure 2 - 4. Block Diagram of VersaLab's Temperature Control Thermal Circuit.

Unlike most cryostat temperature control systems, VersaLab temperature control involves no bulk gas flow. Instead, VersaLab utilizes solid and variable conduction in order to provide variable cooling power to the sample chamber. VersaLab performs gross temperature control by selecting this cooling power to roughly match the ramp condition or stable temperature requested by the user. Standard feedback control using heaters and thermometers provides fine temperature control, allowing controlled ramps and stability to be achieved.

A proprietary thermal switch achieves variable cooling power to the sample chamber by providing a variable thermal conductance between the first stage of the cryocooler and the bottom of the sample chamber as shown in Figure 2-4.

The first stage of the cryocooler will warm up when the sample chamber is cooled because heat is transferred from the chamber to the first stage via the thermal switch. As a result, rapid cooling of the chamber at high temperatures may result in slower cooling at lower temperatures while the system waits for the first stage to cool back down. VersaLab may not be able to maintain linear ramps while cooling at fast cooling rates due to this effect.

2.3.2 Sample Chamber

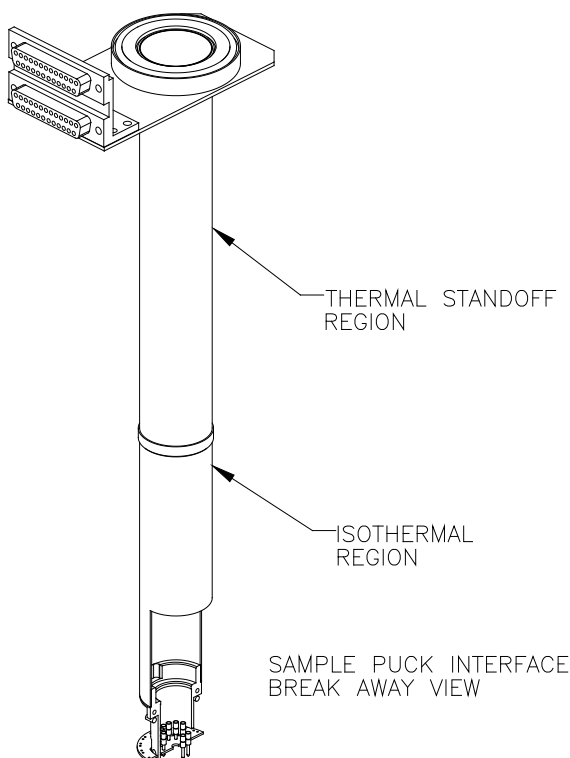


Figure 2 - 5. VersaLab Sample Chamber with Sample Puck Interface shown

The sample chamber consists of three main sections:

- Thermal standoff region. The upper region of the sample chamber is constructed from thin-walled stainless steel tubing, allowing one end to be at room temperature while the other is cooled to 50 K.
- Isothermal region. The lower portion of the sample tube is constructed from high conductivity copper in order to provide a uniform thermal environment for experiments.
- Sample puck interface. At the bottom of the isothermal region is an interface for sample pucks. A cylindrical surface provides a thermal interface to the puck fingers. A 12-pin hermetic electrical feedthrough provides an electrical interface to the puck, and brings the 12 leads out of the sample atmosphere into the vacuum space. See Appendix C.3 for a more detailed description of the sample wiring.

The temperature of the sample puck interface, called the system temperature, is measured using the block thermometer, which is mounted to the bottom of the sample chamber in the cryostat vacuum space. The block thermometer is near the sample puck and therefore represents an accurate estimate of the sample temperature. However, thermal gradients will limit this accuracy during fast slewing of the sample temperature. The most accurate temperature measurement is made when the temperature is stable. When using a standard sample puck, slewing will typically cause a temperature lag of about 20 seconds between the puck and block temperatures. So, slewing at 2 K/minute will yield a 0.7 K temperature error. The value of the lag and error is dependent on sample mounting and the mass of the sample puck.

In case the block thermometer fails, a second thermometer, the backup thermometer, is mounted in an identical location. VersaLab can be configured by Quantum Design to use either the block or backup thermometer to measure the system temperature. If you suspect that either the block or backup thermometers have failed, you can compare their values using the Log Data dialog. Select the menu item “Utilities > Log Data”, and then select the Diagnostics tab. Then select the check boxes for the block and backup thermometers. The reported temperature values should agree to within 0.5 %. (See Appendix B.2 for more details about Log Data.)

Two heaters are mounted to the outside of the sample chamber and are used for temperature control. The block heater is mounted to the bottom of the sample chamber, near the block and backup thermometers. The neck heater is physically larger, and extends from the isothermal region up into the stainless steel region of the sample chamber. These two heaters are used for fine temperature control. When the temperature is stable or nearly so, only the block heater is used. During warming, the neck heater and block heater are used in combination so as to minimize thermal gradients.

2.3.3 Option Temperature Control and Description of Temperature Nomenclature

When no measurement option is in use, VersaLab temperature control uses the system temperature, measured by the block or backup thermometer, as described above. This scheme is appropriate for samples mounted on standard pucks and in good thermal contact with the puck interface. The Electronic Transport Option is a good example of such a measurement. However, some measurement options, such as the Vibrating Sample Magnetometer, place the sample in a position such that it is not in good thermal contact with the puck (and therefore the block and backup thermometers). Therefore, these options provide an additional thermometer near the sample. VersaLab uses this option thermometer in order to provide accurate temperature control and temperature measurement.

The option temperature is usually reported by the measurement option hardware to VersaLab via the CAN bus. For details about measurement option thermometers, refer to the relevant measurement option manual.

For clarity, the three temperatures used by the VersaLab temperature control system are:

- System temperature: represents the puck interface temperature. This is the block or backup temperature, depending on how VersaLab is configured.
- Option temperature: represents the sample temperature for some measurement options. This temperature is read by a measurement option thermometer, reported to VersaLab via the CAN bus.
- Control temperature: the temperature that is being used for feedback control. With no measurement option active, and for some measurement options without option thermometers, the control temperature is the system temperature. For measurement options that request control off of the option thermometer, the control temperature is the option temperature.

2.3.4 Temperature Control Electronics and Firmware

All of the electronic resources for temperature control are provided by the VersaLab controller board located in the electronics tower at the rear of VersaLab. The resources provided are:

- Thermometry. The controller board provides excitation, read back, and table interpolation for the following thermometers. Temperature update rates are 2 Hz on thermometers used for feedback (normally block and switch pot) and 3.5 Hz on other thermometers.
 - Block thermometer. Used for feedback unless system is configured to use backup thermometer or user thermometer.
 - Backup thermometer.
 - User thermometer. Connected to 4-pin Lemo connector on the back of VersaLab. This thermometer is normally used for calibration of block and backup thermometers, but can be used to read out user-provided thermometers.
 - Switch thermometer. Used for feedback control of thermal switch.
 - Diagnostic thermometers: magnet, cryocooler first stage, and cryocooler second stage.
- Heaters. The control electronics drive the following heaters to achieve feedback control of temperature:
 - Block heater, capable of delivering about 20 W.
 - Neck heater, capable of delivering about 20 W.
 - Switch heater.

In addition to providing these hardware resources, the VersaLab controller board provides all temperature control automation by means of the VersaLab firmware on the CPU card of the controller board. The PID feedback control and state machine are both implemented in this firmware. The MultiVu software simply sends commands to and receives status from the VersaLab firmware in order to execute user-requested temperature set points.

2.4 Magnetic Field Control System

2.4.1 Magnet and Leads

The magnetic field in VersaLab is generated by a superconducting niobium-titanium (NbTi) solenoid mounted in the cryostat vacuum space. The magnet generates a vertical magnetic field. The standard magnet generates a maximum 3 T field at approximately 20 A of current. While most magnets are cooled by liquid or gaseous helium, the VersaLab magnet is cooled by solid conduction. Copper straps connect the magnet to the cryocooler second stage in order to cool it.

The VersaLab magnet does not have a superconducting switch. Rather, the magnet controller is directly connected, via magnet leads, to the superconducting solenoid. With no liquid cryogen to boil off due to heat dissipated in the magnet leads, a superconducting switch is not necessary.

The magnet leads inside the cryostat consist of three stages:

- The section between room temperature and the cryocooler first stage, which is constructed of copper wire. The design minimizes the total heat load on the cryocooler first stage considering solid conduction and joule heating at maximum current.
- The section between the cryocooler first and second stages, which is constructed of high temperature superconductor, eliminating joule heating.
- The section between the cryocooler second stage and the magnet, which is constructed of NbTi alloy, also eliminating joule heating.

Low thermal resistance, but electrically isolating thermal anchors, cool the magnet leads at the first and second stages of the cryocooler in order to make sure that the high temperature superconductor and NbTi sections are superconducting and to minimize heat load to the next stage.

2.4.2 Magnet Controller

The VersaLab magnet controller is located in the center rear portion of the electronics tower. The controller provides enough current and voltage to ramp a standard 3 T VersaLab magnet at 300 Oe/sec. Due to an inherently 4-quadrant design and analog current feedback, the system is able to achieve linear field ramps with no discontinuity or kink at zero field and negligible overshoot when stabilizing.

The VersaLab controller board and firmware automate the magnet controller. The controller board ramps the value sent to the magnet controller DAC in order to request current set points for analog feedback and achieve linear field ramps. The control board also reads back values from an ADC in order to determine the voltage across a shunt resistor to measure magnet current. The control board also monitors the magnet thermometer in order to ensure that the magnet is cold enough to operate properly. If the magnet temperature exceeds a critical value (stored in the cryostat SROM), then the magnet current is ramped to zero and the system declares a quench.

2.4.3 Magnetic Field Accuracy, Uniformity and Magnet Reset

VersaLab reports the magnetic field as the measured magnet current multiplied by a calibration factor determined at the factory for your magnet. (This factor is stored in the cryostat SROM.) The system does not take magnet relaxation or trapped flux into account. For information on the resulting field remanence, see your system specifications.

The field remanence can be reduced to a value near the earth's field by performing a magnet reset. See Section 4.4.3 for information about this procedure.

The uniform region of the magnetic field is centered 4.0 cm (1.6 inches) above the top surface of a standard sample puck. See Figure 2-7. The field is uniform to within $\pm 0.1\%$ over a 2.5 cm axial length centered at that location. VersaLab is calibrated to report the magnetic field at the center of the uniform region. The magnetic field at the puck surface is up to 1% lower than the value reported by VersaLab, because the puck surface is not in the uniform region.

2.4.4 Magnetic Shield

A magnetic shield surrounds the VersaLab cryostat in order to reduce the field value experienced outside the system. The field is below 5 gauss everywhere outside the VersaLab enclosure except near the top and corners of the enclosure top tray. The field is below 5 gauss for any point at least 5 cm (2 inches) away from the top tray.

The magnetic shield also serves to protect sensitive experiments from external magnetic field sources. For example, the shield protects the Vibrating Sample Magnetometer coil set and coil set leads from magnetic signals originating in the VSM Linear Motor. The shield can also protect experiments from various environmental sources that may exist in your laboratory, such as other instruments that generate fields and magnetic building elements.

2.5 Chamber Atmosphere Control System

Many experiments carried out in VersaLab require control of the experimental atmosphere present in the sample chamber. Any experiment that involves low temperatures requires an atmosphere that will not freeze or condense on the sample and other experimental hardware. Some experiments benefit from the thermal uniformity provided by exchange gas, while others require thermal isolation provided by high vacuum. VersaLab meets all of these needs with its chamber atmosphere control system. In addition, this system enables VersaLab to pump out the cryostat vacuum case prior to cooling down.

The chamber atmosphere control system consists of the following components, which are described in more detail in the sections below:

- Diaphragm pump, which is used to pump out the sample chamber to pressures as low as 2 Torr.
- Gas handling manifold, which contains hardware to control and measure chamber gas. Hardware mounted to the manifold includes:
 - Pressure gauge
 - Solenoid valves
 - High vacuum valve

- KF flange and baffle set
- Cryopump, which is used to pump the chamber out to high vacuum.
- Vent gas, which is used to vent up the sample chamber and reduce atmospheric contaminants in the chamber.
- VersaLab controller board, which automates the pump and valve sequencing needed for chamber operations.

2.5.1 Diaphragm Pump

The diaphragm pump is capable of achieving pressures below 2 Torr. It is used to:

- Evacuate the sample chamber.
- Pump on the cryopump during regeneration.
- Evacuate the cryostat vacuum case before cooling down a warm VersaLab system.

The rubber diaphragms in the diaphragm pump have a lifetime of approximately 10,000 running hours. Because the diaphragm pump does not run continuously in VersaLab, the diaphragms are expected to last for many years. Normally, the diaphragm pump will be rebuilt at every other cryocooler maintenance (see Appendix A.4). If you suspect that your diaphragm pump has worn out and requires maintenance, please contact Quantum Design.

2.5.2 Gas Handling Manifold

The gas handling manifold, which is bolted to the cryostat top plate under the magnetic shield, provides most of the gas handling. Three solenoid valves (the vent valve, pump valve, and case valve) are mounted to the manifold. The high vacuum valve, a linearly actuated bellows valve, provides a high conductance connection between the chamber and cryopump when open. The pressure gauge monitors the pressure in the sample chamber. When the system is pumping on the vacuum case, the pressure gauge can also monitor the pressure in the case.

2.5.3 KF Flange and Baffle Set

The sample chamber is accessed through the KF-40 flange mounted to the gas handling manifold. In order to seal the flange and prevent thermal radiation from reaching your sample, you use the baffle set (Figure 2-6). In order to prevent leaks into the sample chamber, be sure to keep the sealing surfaces of the KF flange and baffle set clean. Also, be sure not to damage the O-ring used to seal this flange.

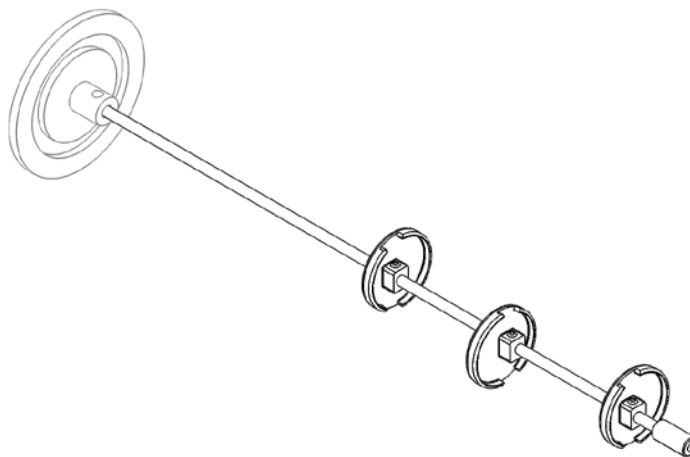


Figure 2 - 6. Baffle Set

A threaded adapter is present on the end of the rod in order to allow the attachment of accessories, such as the contact baffle used with the Heat Capacity and Thermal Transport Options.

2.5.4 Cryopump

The cryopump is used to achieve high vacuum in the sample chamber, typically $< 10^{-4}$ Torr. The cryopump is permanently mounted in the cryostat vacuum case. Therefore, hardware is provided to allow complete regeneration of the cryopump in situ while the system is cold.

The cryopump may periodically need regeneration due to the build up of contaminant gases and adsorbed helium. When this happens, the cryopump needs to be heated up and pumped out in order to remove the condensed and adsorbed gases, a process called regeneration. It is equipped with a heater to reach room temperature while the system is cold without excessive warming of the cryostat. See Section 4.4.4 for details about this procedure.

You prevent the need to regenerate the cryopump often by keeping the sample chamber clean. Avoid placing wet objects, such as a cold baffle set that has condensed water on it, in the sample chamber. Be sure to purge and seal the sample chamber before attempting to set high vacuum.

In order to ensure that the cryopump stages are clean before cooling down a warm system, the cool down procedure involves regenerating the cryopump at room temperature before starting the cool down. See Section 4.3 for details.


2.5.5 Vent Gas and Regulator

Unlike conventional cryostats, VersaLab does not have dewar boil off gas available for venting the sample chamber. Therefore, you must supply a bottle of vent gas. A regulator on the vent gas bottle sets an appropriate pressure for venting the sample chamber. The vent gas is connected to VersaLab at the bottom rear of the base. See Section 4.2.2 for details about choice of vent gas and connection instructions.

2.5.6 Chamber Operations

VersaLab supports several different chamber operations, described below. It is important to note that any chamber operation involving pumping should be performed only when the sample chamber KF flange is sealed. Otherwise, the chamber will not be pumped down as desired during these operations.

Before setting a temperature below 295 K, you should always be sure that the sample chamber is sealed and contains only clean gas. Normally you do this by executing a purge and seal operation after sealing the KF flange at room temperature. Failure to do so can cause contaminants to freeze inside the sample chamber, causing problems with many experiments. Measurement options provide sample installation wizards that perform chamber operations at the appropriate times when installing samples.

WARNING!	
	Be sure to properly seal the sample chamber KF flange before setting temperatures below 295 K. Failure to do so will cause ice to build up in the chamber, a condition that is hazardous and may damage the VersaLab sample chamber due to excessive pressures resulting from an ice plug. Even a small leak can cause ice to build up, so be sure to keep the KF flange surfaces clean and to not damage the O-ring used to seal the flange.

The VersaLab chamber operations are described below:

- **Seal.** Sealing the chamber simply closes all valves, leaving the chamber atmosphere unchanged.
- **Purge and Seal.** Purging and sealing the sample chamber performs four cycles of pumping out most of the chamber gas and then filling it up with vent gas. Finally, the chamber is pumped down to a pressure that is appropriate for experiments that require exchange gas, about 10 Torr. Normally, you purge and seal the chamber at room temperature after installing a sample in order to clean out atmospheric contaminants introduced while the chamber was open.
- **Vent and Seal.** Venting and sealing the sample chamber fills the chamber with vent gas, and then closes the vent valve. This operation is useful when you want to conserve vent gas, because vent gas will not flow out of the vent gas supply bottle once the vent valve is closed. You can use this operation before opening up the sample chamber at room temperature in order to change samples.
- **Pump Continuous.** This operation uses the diaphragm pump to evacuate the sample chamber. The pump valve remains open and the diaphragm pump remains on indefinitely. The lifetime of the diaphragm pump is shortened if the system is left pumping continuously for extended periods.
- **Vent Continuous.** This operation, also known as flooding, fills the chamber with vent gas, and leaves the vent valve open. Because the flow of vent gas is continuous, this operation minimizes the amount of air that enters the chamber. To ensure that the vent gas bottle is not inadvertently emptied, the vent valve will automatically seal after two minutes.
- **High Vacuum.** This operation uses the cryopump to pump a high vacuum, typically $< 10^{-4}$ Torr, in the sample chamber. First, the diaphragm pump evacuates the sample chamber to < 2 Torr. Then the high vacuum valve is opened, exposing the sample chamber to the cryopump. The system declares high vacuum when the pressure gets below 10^{-4} Torr.

2.6 Measurement System

2.6.1 Sample Pucks and Assorted Tools

The sample puck is a unique modular component that gives VersaLab great flexibility. The puck is the sample holder for many experiments that do not require sample motion or thermal isolation of the sample. The Electronic Transport Option is a good example. Even when the sample is not mounted directly to the puck surface, measurement hardware is mounted to a puck. In the Vibrating Sample Magnetometer, the detection coil set is mounted to a puck. In the Heat Capacity Option, the heat capacity platform and leads are mounted to a puck.

The puck is a 2.3 cm (0.91 inch) diameter disk constructed of high conductivity copper that maintains high thermal uniformity. The puck is gold plated to prevent oxidation, providing a surface on which samples can be mounted with good thermal contact. The system thermometers (see Section 2.3.2) are mounted directly beneath the installed puck, providing good contact between sample and thermometers. Measurement options that use alternate sample mounting methods provide a thermometer close to the sample.

The base of the puck contains 12 solder pads to allow connection of the sample (or other measurement option hardware) to the experimental sample wiring. These solder pads are connected to a set of 12 sockets on the base of the puck, which contact pins in the bottom of the sample chamber. The puck is keyed to ensure that the electrical connectors properly align. See Section 2.6.5 for details on experimental wiring. See Appendix C for an experimental wiring pin out diagram.

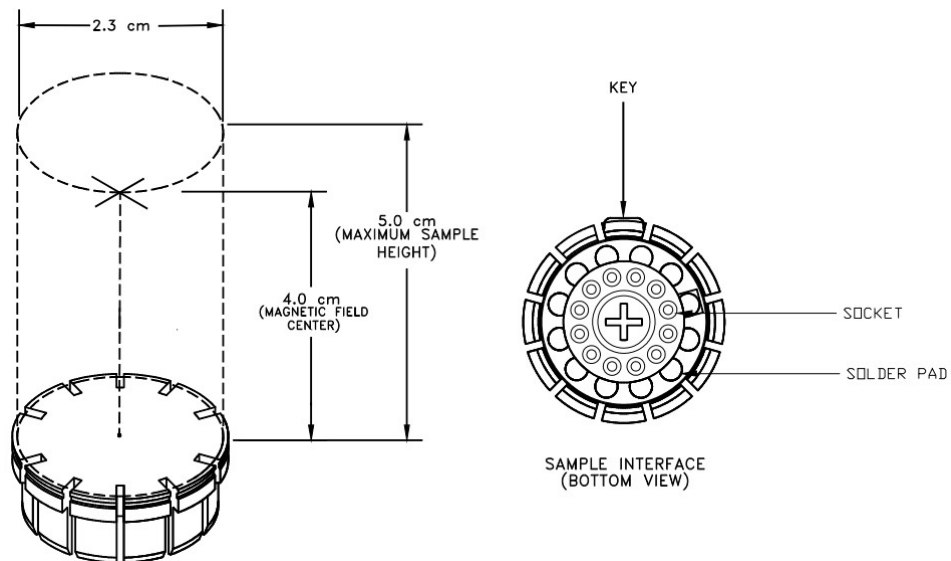


Figure 2 - 7. Top and Bottom views of a Sample Puck

2.6.2 Sample Insertion Tool

The puck-insertion tool is a long rod used for installing the puck in the sample space. The lever of the puck-insertion tool is engaged when it is lying flat across the handle, as is shown in Figure 1-2. When the lever is engaged, the tool grips the puck by a groove in its outer rim.

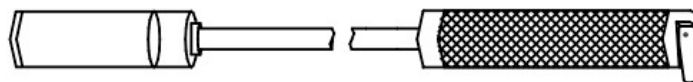



Figure 2 - 8. Sample Insertion Tool with Lever in Engaged Position

The puck-insertion procedure is described in Section 1.3.2.1.

	<p>WARNING!</p> <p>Read section 1.3.2.1 before attempting to insert a puck into a VersaLab. Failure to follow the directions correctly could cause damage to the instrument.</p>
---	---

2.6.3 Puck Adjustment Tool

The puck adjustment tool (Figure 2-9) is used to adjust the position of the fingers of the puck so that the fingers maintain solid thermal contact with the thermal interface located at the bottom of the sample chamber. Use the puck adjustment tool after you have inserted the sample puck into the sample chamber approximately 10 times or whenever the puck fits loosely into the bottom of the chamber. Instructions for using the puck-adjustment tool are given in Section A-2.

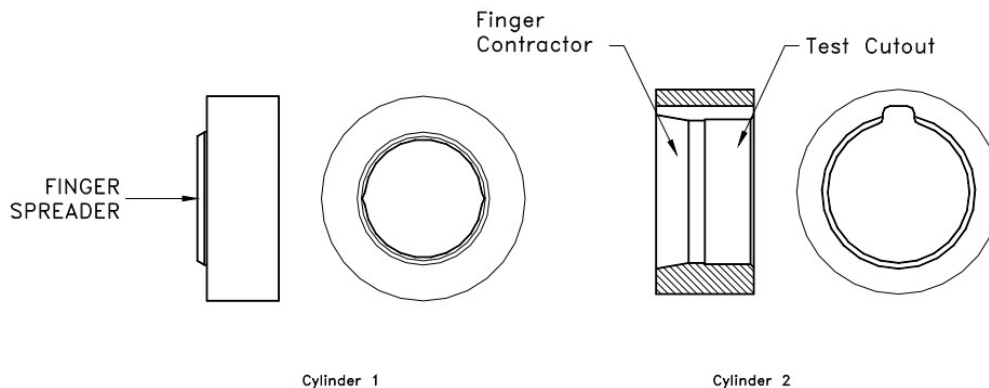


Figure 2 - 9. Puck Adjustment Tool

2.6.4 Puck Wiring Test Station

The puck-wiring test station (Figure 2-10) is used to verify the integrity of the wiring between a sample and puck or the wiring of any option hardware present on a puck. The test station contains three sets of contacts, all wired in parallel: a Lemo connector identical to the sample chamber connector on the probe head, a puck connector, and 12 banana jacks. VersaLab measurement options come with templates that label the banana jack functions when the standard cabling is being used for an option.

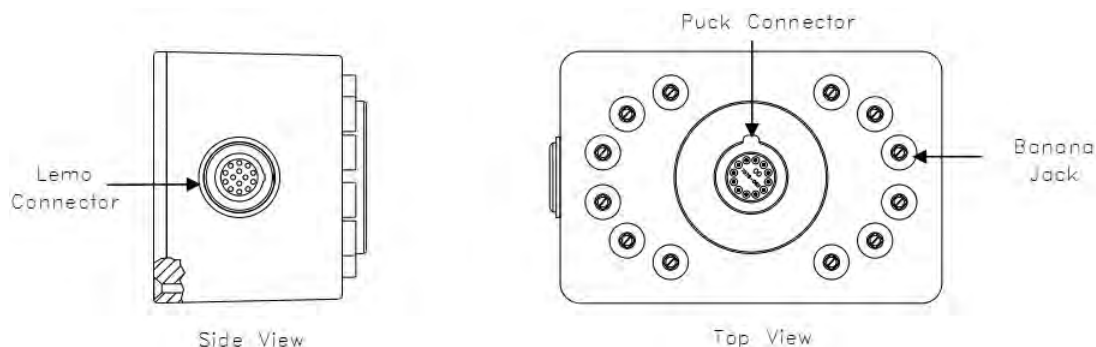


Figure 2 - 10. Puck Wiring Test Station

2.6.5 Experimental Wiring

The experimental wiring in VersaLab consists of the following components:

- The Lemo connector under the VersaLab top cover.
- A cable between the Lemo connector and the top of the sample chamber. A magnetic shield protects this cable from magnetic signal interference
- The cryogenic sample chamber wiring, described in more detail below.
- The vacuum tight electrical feedthrough at the bottom of the sample chamber. The sample puck plugs into the pins at the bottom of the sample chamber provided by this feedthrough.

2.6.6 Chamber Wiring Current Limits

In order to minimize heat leak down the sample chamber while allowing high sample currents to be used, two types of sample wires are used. Four of six pairs of sample wires are designed to minimize heat leak, and can carry a maximum of 10 mA without excessive sample chamber heating or damage to the wires. Two of the six pairs are designed to carry high currents, and can carry 0.5 A continuously without excessive sample chamber heating or damage to the wires. The low current pairs are connected to pins 3-4, 7-10, and 13-14 on the puck and Lemo connector. The high current pairs are connected to pins 5-6 and 11-12 on the puck and Lemo connector. If you perform an experiment that requires high currents, be sure to connect the high current leads to these pins. See Appendix C.3 for detailed pin out diagrams.

2.6.7 CAN Modules

Measurement electronics are contained within modular electronic housings, commonly called CAN modules. A typical CAN module is shown in Figure 2-11. Connections to measurement hardware are made on the front panel, which is shown in the figure.

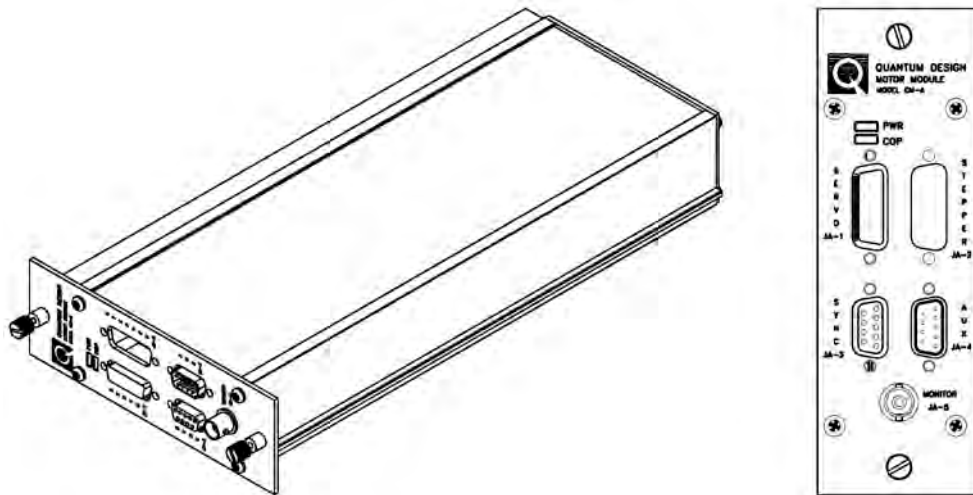


Figure 2 - 11. Model CM-A VSM Motor Module

The 9-pin QD-CAN D-shell connector on the bottom panel (Figure 2-12) of the modules connects to power, CAN, and synchronization lines in the Module Bay, described below. One or two guide holes are present, depending on whether the module dissipates high or low power. All slots in the VersaLab Module Bay accept high power modules.

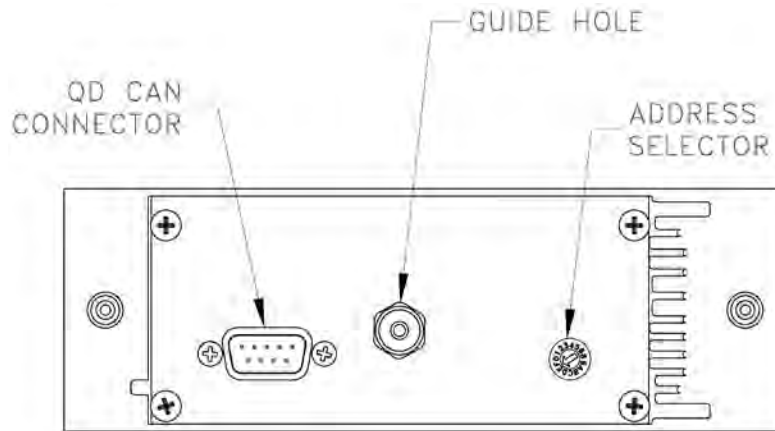


Figure 2 - 12. Rear Panel of the Model CM-A VSM Motor Module

CAN modules communicate with VersaLab, other CAN modules and the CANOpen-USB dongle via the CAN bus. The PC software communicates with CAN modules using CAN over USB via the CANOpen-USB dongle.

For a detailed description of specific CAN modules, consult the relevant measurement option manual. For instructions on insertion and removal of CAN modules, see Section 3.2.5 (in Option setup).

2.6.8 Module Bay

CAN Modules reside in the Module Bay, located under the plastic lid in the VersaLab electronics tower. The Module Bay provides power, CAN, and synchronization lines to modules. Any module may be inserted into any slot in the Module Bay, because all slots are high power slots.



Figure 2 - 13. CAN Module Bay shown with modules installed

The Module Bay also supplies cooling air to the modules. In order to ensure proper cooling, be sure to cover any unused slots with the covers provided with your system.



WARNING!

Always cover any unused slots in the Module Bay. Failure to do so may result in overheating of modules. Uncovered slots also increase the risk of foreign objects falling into the module bay, possibly damaging sensitive electronics due to electrical shorts. Take care to avoid dropping any objects into the Module Bay.

C H A P T E R 3

Software

3.1 Overview

This chapter describes MultiVu, the Windows™ software that allows user control of the operation of the VersaLab hardware. MultiVu combines, in a single user interface, the basic instrument control, status reporting, and data collection (section 3.3); measurement options (section 3.4); sequence editing and sequence execution (section 3.5); and graphing (section 3.6). In addition to these features, MultiVu also contains instrument utilities, diagnostics, and error reporting. Instructions for installing VersaLab MultiVu are given in section 3.2.

3.2 Installing VersaLab MultiVu

MultiVu may be freely installed on any computer. To install MultiVu, simply run the “Versalab Setup.exe” program and follow the instructions provided within the setup program.



Figure 3-1. VersaLab MultiVu Setup Wizard main page

Note If you are upgrading VersaLab MultiVu from an older installation package, system calibrations (*.cal), configuration files (*.cfg), SROM, and user data should be backed up prior to using the latest MultiVu installer. Also be sure to back up any calibrations for installed options. Once all calibrations, configurations files, SROM, and data have been saved, the older version of VersaLab MultiVu should be removed by launching the uninstaller through the computer's Control Panel.

3.2.1 Full Installation

To install MultiVu on a computer that is connected to your VersaLab system, select "Full Installation" from the popup list in the "Select Components" step. Depending on the options that are present in your system, you may also want to enable some of the option installers (Figure 3-2).

Note The CAN sub-system must be installed prior to installing VersaLab MultiVu (Section 3.2.2).

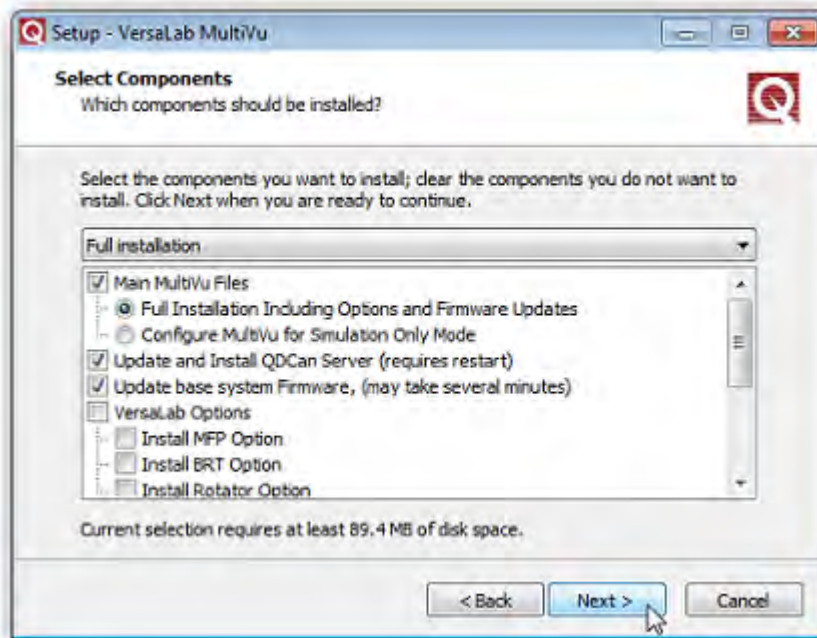



Figure 3-2. Components Selection Dialog - Full Installation

Note VersaLab MultiVu will flash modules that require firmware. Before beginning the full install, it is important remember that the VersaLab has only four module bays; therefore only modules that are in the module bays and options that do not require firmware will be available during the installation. If you have more than four modules for your VersaLab system, the installer will need to be run a second time.



WARNING!

Inserting or removing CAN Modules with the power ON may permanently damage the electronics. Always turn OFF VersaLab when inserting or removing modules.

3.2.2 QdCAN Installation

Before initiating the MultiVu installation (as described for the Full Installation above), the installer will check the computer for the QdCAN directory. A warning will be issued if QdCAN cannot be located (Figure 3-3), along with the steps carried out in the CAN-only installation.

Note Be sure to UNPLUG the CAN USB dongle from the computer before completing the QdCAN installation.

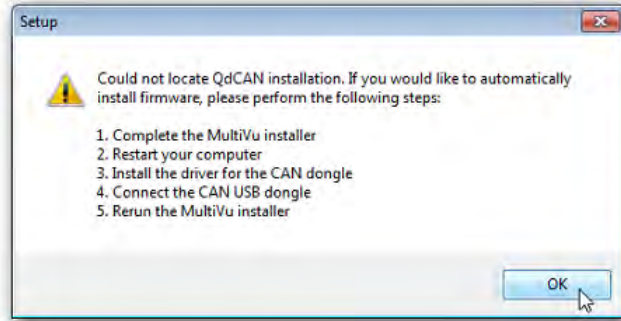


Figure 3-3. QdCAN setup window

After clicking “OK” on the CAN-setup window, the installer window will show only QdCAN as being checked in the list of items to install. Once the installation has completed, restart your computer. Navigate to your computer’s **Control Panel > Device Manager** and PLUG-IN the CAN USB dongle. After a few seconds, QdCAN should appear in the **Device Manager** list. At this point the installer can be rerun for a **Full**, **Custom**, or **Simulation** installation of MultiVu and firmware.

3.2.3 Custom Installation

Custom installations may also be performed on a computer connected to your VersaLab system. To do so, select “Custom Installation” from the pull-down list in the “Select Components” step. Depending on the options that are present in your system, you may also want to enable some of the option installers (see Figure 3-4).

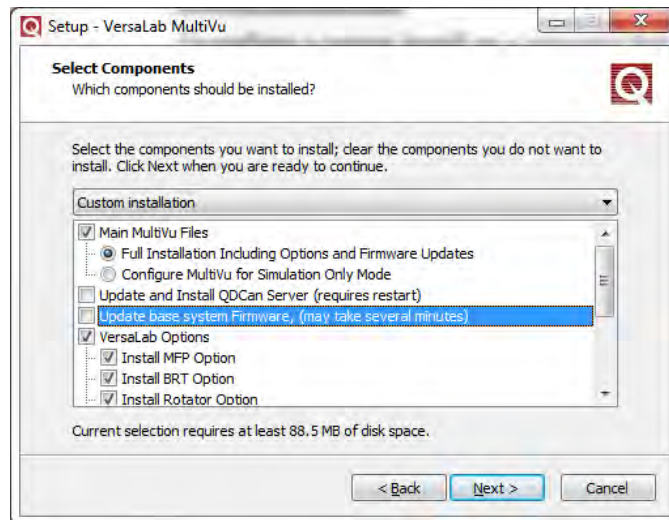


Figure 3-4. Components Selection Dialog for Custom MultiVu Installation

3.2.4 Simulation Installation

To install MultiVu on a computer that is not connected to your VersaLab 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 3-5).

Note Options selected during this installation mode will run as simulation when activated.

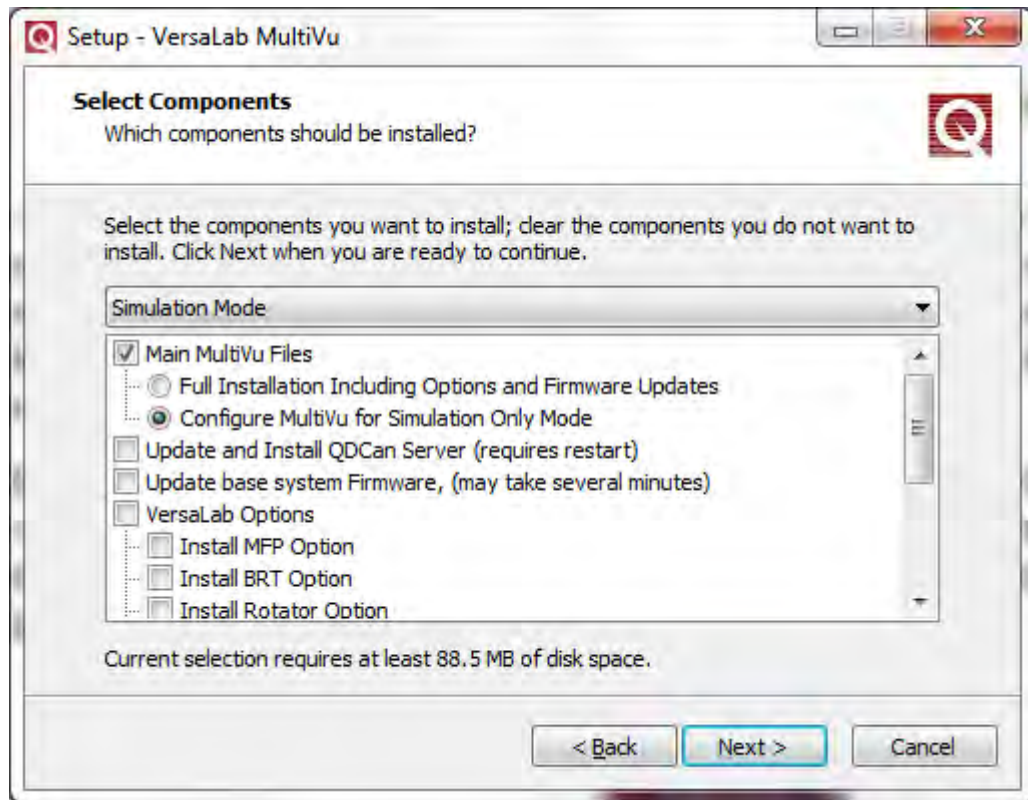



Figure 3-5. Components Selection Dialog for Simulation Mode Setup

Once installed, use the desktop icon for running MultiVu in simulation mode, which works without VersaLab 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 on office computers, and is not intended for use on the VersaLab computer.

3.2.5 Installing CAN Modules

You may need to install optional CAN modules in the VersaLab Module Bay before using a measurement option. If the modules are already installed, you can leave them there, even if they are not in use. If you need to insert modules, follow these steps:

1. It is necessary to turn OFF VersaLab before inserting a module.

	WARNING!
	Inserting or removing CAN Modules with the power ON may permanently damage the electronics. Always turn OFF VersaLab when inserting or removing modules.

2. Remove the cover from the module slot where you intend to insert the module.
3. Carefully slide the module into the slot. The module should be oriented such that the bottom of the text faces the front of VersaLab. You will feel the 9-pin D-shell connector on the module mate with the module bay.
4. Once the module front panel is flush with the module bay, securely hand-tighten the screws on the front panel to secure the module and properly ground its chassis.
5. Turn the power to VersaLab ON. Wait for it to boot. It may be necessary to restart MultiVu and QDCAN Manager.

CAUTION!
Leaving VersaLab's power OFF for longer than 10 minutes may cause the system to warm up irreversibly. If this happens, another full cool down will be required. Try to minimize the amount of time where VersaLab's power is off.

After installing any modules, install the rest of the measurement option hardware as instructed in the option manual. Then activate the option using the MultiVu menu item "Utilities > Activate Option..." Select the desired option in the Option Manager window and click "Activate -->>."

The option software will now appear. This software provides many functions, including a wizard for inserting and removing samples, commands for performing immediate-mode measurements, and sequence commands for automating measurements. Refer to the option manual for details.

3.2.6 Starting MultiVu

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

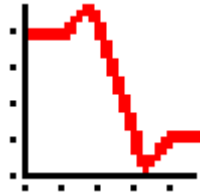


Figure 3-6. VersaLab MultiVu Icon

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

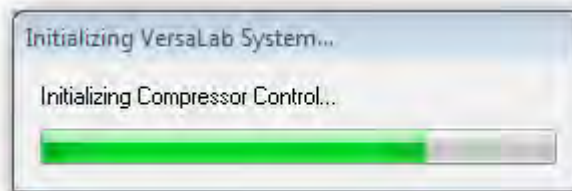


Figure 3-7. System Start-up Progress Dialog

When the MultiVu software is not running, VersaLab goes into a standby state to prevent equipment damage. Most users will leave MultiVu running on the VersaLab computer at all times.

You can monitor the system status when MultiVu is running, so Quantum Design recommends that you leave MultiVu running even when you are not using the instrument but it is cold. If you warm up the instrument, quit MultiVu before turning the system off.

3.2.7 MultiVu Directories and Files

Table 3-1 on the following page lists the directories and descriptions of files associated with the VersaLab MultiVu software.

Table 3-1. VersaLab MultiVu Directories and Files

DIRECTORY	FILES	DESCRIPTION	MANUAL REFERENCE
C:\QdVersaLab\		Root directory containing all files related to VersaLab MultiVu	
C:\ QdVersaLab\Data		Default directory to store all measurement data	
C:\ QdVersaLab\Macros		Directory for basic scripts	3.4.2
C:\ QdVersaLab\MultiVu	VersaLab.exe	VersaLab MultiVu program file	
	*.dll	Libraries required for VersaLab MultiVu (list of files depends on installed options)	
	*.chm	On-line manuals	
	Event.log	Log file containing diagnostic information for trouble shooting	
	BKG.dat	Data log that continuously runs in the background of the MultiVu program. Useful for diagnostic purposes.	
	vcredist_x86.exe	Microsoft Visual C++ setup	
C:\QdVersaLab\Qmaps	*.qmap	Definition files for additional data logging	Appendix B.2.3
C:\QdVersaLab\Sequence		Default directory to store sequences	3.4
C:\ QdVersaLab\Tools	Export Data.exe	Utility to export data for analysis and post processing.	
C:\QdVersaLab\Wizards	*.bas *.obm	Various script-based utilities (and associated files) which guide the user through specific system functions.	3.2.9

3.3 Graphical User Interface

The main MultiVu window is shown in the figure below. This window displays essential status information about the instrument and allows control of the instrument with simple mouse click commands. The tool bar, control center, sequence command bar, and status bar may each be docked and un-docked by double-clicking any non-control region or by dragging them around the main window. They may also each be hidden using the “View” menu.

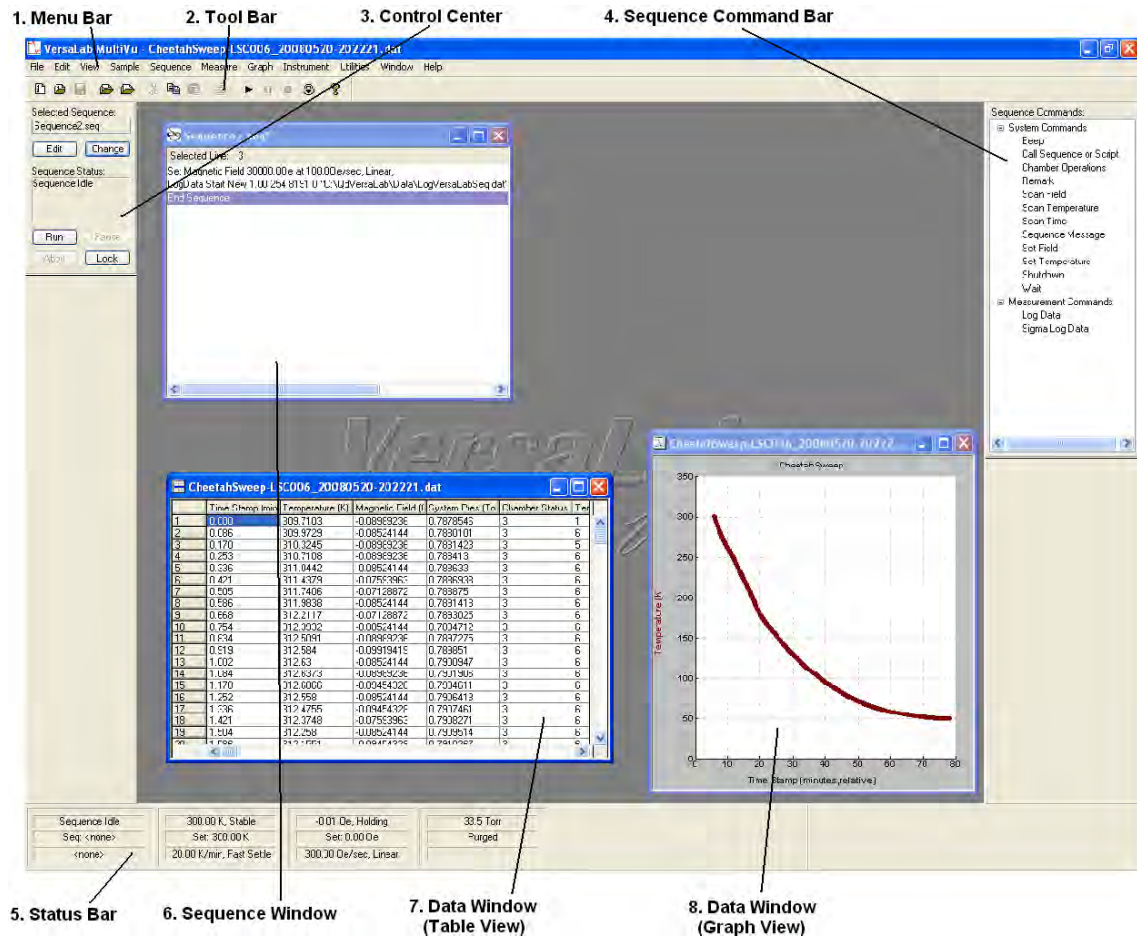


Figure 3-8. MultiVu Main Window

3.3.1 Menu Bar

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

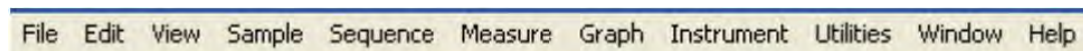


Figure 3-9. Main Menu Bar

- 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. This menu is populated only when a measurement option is active.
- 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 measurements executed by measurement options. This menu is populated only when a measurement option is active.
- 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 atmosphere state of the sample chamber, and to put the instrument into standby mode when unused.
- UTILITIES - Utilities menu contains commands to activate and configure measurement options, and to help maintain and troubleshoot the instrument, including cooling the system down, regenerating the cryopump, 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 VersaLab, and access to the user documentation.

3.3.2 Tool Bar

The Tool Bar contains shortcuts for common menu items.

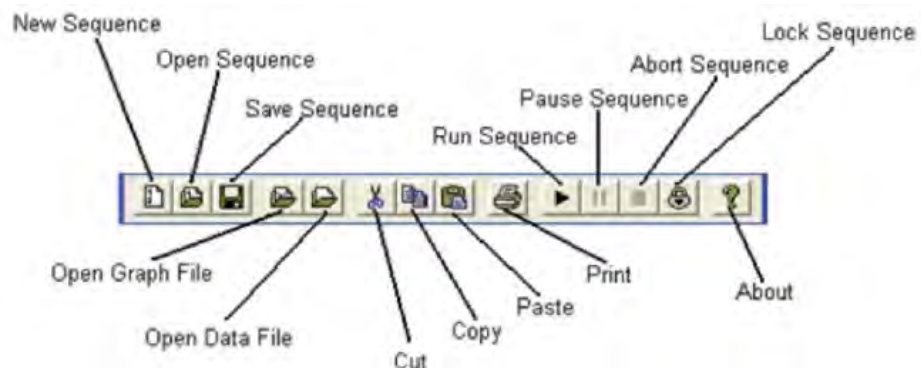


Figure 3-10. Tool Bar

3.3.3 Control Center

The Control Center (Figure 3-11) shows the currently selected sequence file name and sequence execution status. In addition, there are buttons to change the selected sequence and edit, run, pause, abort, or lock the selected sequence.

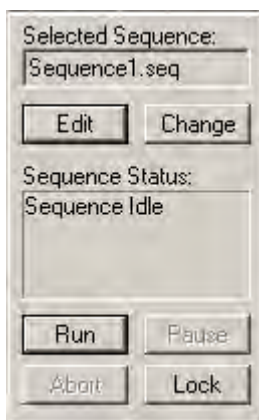


Figure 3-11. Sequence Control Center

3.3.4 Sequence Command Bar

The Sequence Command Bar (Figure 3-12) 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. Measurement option sequence commands are present in the "Measurement Commands" branch when a measurement option is active. More on sequences is found in the section below.

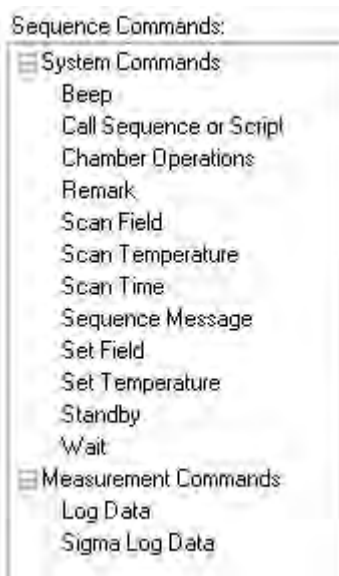
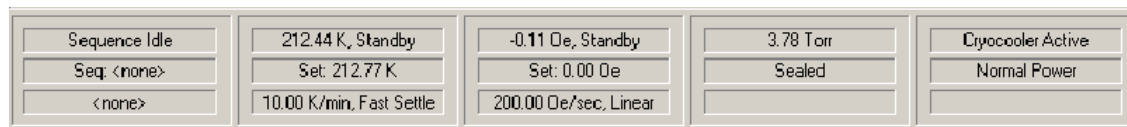


Figure 3-12. Sequence Command Bar

3.3.5 Status Bar

The Status Bar contains information about the status of the instrument. Click on the panels in the status bar to access dialogs for setting temperature, magnetic field, and chamber atmosphere actions.



<u>Sequence:</u>	<u>Temperature:</u>	<u>Magnetic Field:</u>	<u>Sample Chamber:</u>	<u>Cryocooler:</u>
-status	-present state	-present state	-pressure	-status
-file name	-setpoint	-setpoint	-state	-cooling power
-execution line	-rate and mode	-rate and mode		

Figure 3-13. MultiVu's VersaLab Status Bar

3.3.6 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.



Figure 3-14. Run Button

3.3.7 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 spreadsheets and other graphing programs for additional analysis using the standard Windows™ copy and paste features.

3.3.8 Data Window (Graph View)

The Data Window (Graph View) shows a data file in graph format. Each point on the graph represents a single data record. A data record consists of several data items collected simultaneously, such as sample temperature, magnetic field, and magnetic moment. You may view a single data record in its own window by double-clicking any record in the graph view. The record view and graph view are linked, so that the highlighted point on the graph view corresponds to the record shown in the record view. The appearance of the graph view may be manipulated extensively and templates may be applied to the graph view so that you do not need to set each graph characteristic individually. To learn more about manipulating the graph view, see Section 3.6.

3.3.9 Instrument Utility Wizards

Under the menu item “Utilities > Wizards >” you access utilities for performing tasks including cryopump maintenance, cooling down the system, and resetting the magnet. When you start a wizard, a dialog box opens and shows instructions to guide you through the process of executing the utility. Follow the on-screen instructions, and consult the relevant section in Chapter 4: System Operation for more details.

3.4 Measurement Options

Most measurements in VersaLab require the use of measurement option software. You do not use a measurement option if you provide the measurement electronics, but all measurements provided by Quantum Design require option software to be active. For details on the process of setting up a measurement option, see the relevant option manual.

In order to activate an option, first set up the option hardware as described in the option manual. Then select the menu item “Utilities > Activate Option...” You will be presented with a dialog showing available measurement options. Select the desired option and press the “Activate -->>” button. The option software will start within the MultiVu window, and will initialize the option hardware. The measurement option will display a control center from which you can:

- Configure the option
- Insert and remove samples
- Set up data files
- Perform immediate-mode (not sequenced) measurements

In order to perform sequenced measurements, you write and execute a sequence in MultiVu as described in Section 3.5.

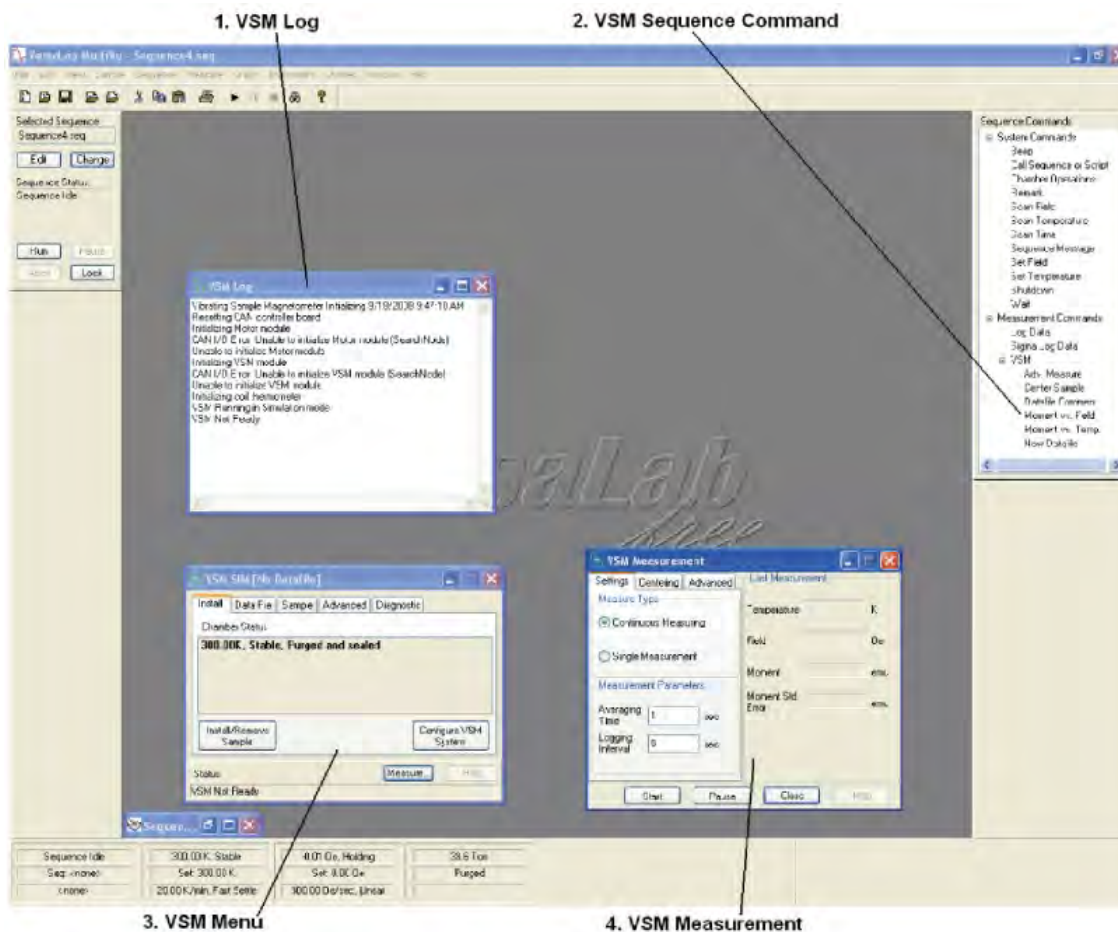





Figure 3-15. VSM Option Control Centers

3.5 Sequences

Operation of VersaLab is automated using sequences. Sequences are simple computer programs instructing the instrument to carry out a number of operations in a predetermined order. Simple looping is supported in sequences, but branching is not. Operations typically found in a sequence file include changing the sample temperature, changing the magnetic field, and measuring some property of the sample (depending on which measurement option is active). Additionally, commands may be placed in sequences to log diagnostic data, 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.

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

3.5.1 Editing Sequences

- Create a new sequence file by clicking “New Sequence”  or selecting the menu item “File > New Sequence.”
- Open an existing sequence file for editing by clicking “Open Sequence”  or selecting the menu item “File > Open... > Sequence.” You will be prompted to locate the file you want to open.
- The top of the sequence window shows the sequence file name. An asterisk symbol (*) after the file name means the sequence has changed since last time the file was saved. Sequences must be saved before you run them.
- Save a sequence file by clicking “Save Sequence”  or selecting the menu item “File Save.” If not already assigned, you will be prompted for a location and file name.
- Add a command to the selected sequence by double-clicking the command in the sequence command bar. The new command is inserted above the highlighted command in the selected sequence window. You will first be presented with a pop-up window to fill in command-specific information. Then a summary of the command will appear in the sequence window. More detail about each available sequence command is found at the end of this section.
- Remove and reorganize sequence commands by highlighting them in the sequence window and using the “Cut,” “Copy,” “Paste,” “Delete,” and “Undo” commands in the “Edit” menu.
- Disable commands in a sequence file without deleting them using “Edit > Disable.” Enable a disabled command with “Edit > Enable” (Figure 3-16). 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.

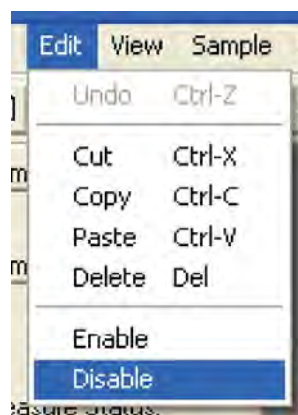


Figure 3-16. Disabling Sequence Commands


3.5.2 Advanced Script Editing (Macros)




Before running sequences, MultiVu automatically compiles sequence files into Visual Basic for Applications (VBA) scripts. 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). VBA scripts give the user the ability to perform branching and more sophisticated loops than standard sequences. Also, communication with third-party instruments via GPIB is possible in VBA scripts. (You must supply GPIB hardware.)

However, Quantum Design cannot certify the instrument behavior when it is automated with user-designed scripts. Many checks and safeguards can be bypassed when running a VBA 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”.

3.5.3 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.

3.5.4 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.
- 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 and does not protect against malicious behavior.
- The instrument does not need to be running a sequence to lock sequence execution.
- Lock sequence execution by clicking “Lock Sequence” , selecting 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.
- Unlock the sequence by clicking “Lock Sequence”  again, selecting 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.

3.5.5 Sequence Commands

The following sequence commands are present in VersaLab with no measurement option activated. Additional measurement commands are available when using an option.

3.5.5.1 SYSTEM COMMANDS

- **Beep** – Causes the computer to make the Windows default beep sound.
- **Call Sequence** – Suspends execution of parent sequence file and begins execution of selected child sequence file or script file. When execution of child sequence file or script is complete, execution of parent sequence will continue with the next line.
- **Chamber Operations** – Changes the state of the sample chamber atmosphere. (Figure. 3-17). See section 2.5.6 for a description of chamber operations.

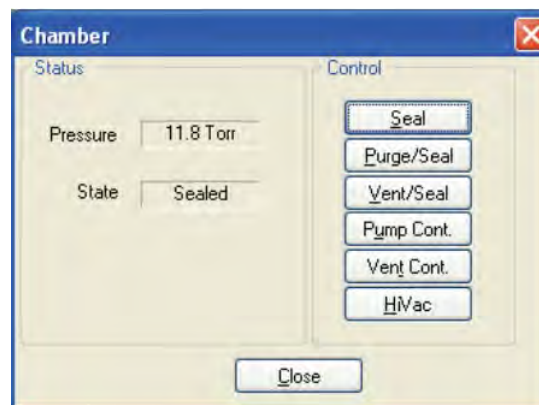


Figure 3-17. Sample Chamber Atmosphere Control Center

- **Remark** – Serves as a message, comment, or visual break for the user only. Does nothing during sequence execution.
- **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^2 , $H^{1/2}$, $1/H$, $\log(H)$). Also set the total number of field steps. (For uniform linear spacing, you may alternatively set the field increment.) Finally, specify the rate and approach mode used by the magnet controller to achieve each set point:
 - Linear: Controller will drive directly to each field and attempt to maintain the specified charging rate as closely as possible until each set point is reached. At each set point the field will stabilize until the commands within the loop are completed.
 - Oscillate: Controller will intentionally overshoot each set point by 70% of the total field change, at the desired rate, and will then oscillate into the set point field in smaller and smaller overshooting steps. At each set point the field will stabilize until the commands within the loop are completed. This is intended to eliminate flux motion in the superconducting magnet windings, yielding a very stable magnetic field and reduced remanence.
 - 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 ramp while they execute.
- **Scan Temperature** – Creates a program loop for executing repeated commands at user-defined temperature increments. All commands between the “Scan Temperature...” line in the sequence and the “End Scan” line in the sequence will be repeated at each temperature specified by the scan temperature command. Set the initial and final temperatures and the scale on which the spacing of the temperature steps should appear uniform (linear, $1/T$, $\log(T)$.) Also set the total number of temperature steps. (For uniform linear spacing, you may alternatively set the temperature increment.) Finally, specify the rate and approach mode used by the temperature controller to achieve each set point:
 - Fast: Controller will drive directly to each temperature and attempt to maintain the specified sweep rate as closely as possible until each set point is reached. At each set point the temperature will stabilize until the commands within the loop are completed. A small temperature overshoot can occur in this mode.
 - No Overshoot: Controller will drive to each set point at the desired rate until it is close to the set point, and will then slow the rate to avoid temperature overshoot. At each set point the temperature will stabilize until the commands within the loop are completed. This is intended for use with highly temperature-hysteretic samples.
 - Sweep: Controller will drive directly from the initial temperature to the final temperature without stopping. Each time a temperature increment defined by the command is reached, the commands inside the scan temperature loop will be executed, but the temperature will continue to change while they execute.
- **Scan Time** – Creates a program loop for executing repeated commands at user-defined time increments (or immediate repetitions with no time increment.) All commands between the “Scan Time...” line in the sequence and the “End Scan” line in the sequence will be repeated at each time specified by the scan time command. Set the total time in seconds and specify whether the spacing of events should be uniform in time or logarithmic in time. And specify the number of steps. This is the number of times the loop will be repeated. (For uniform spacing in time, you may alternatively specify the time increment.) If the total time is set to zero seconds, then the number of steps defines how many times the loop will be repeated in rapid succession.

- **Sequence Message** – Displays a message on the computer screen and pauses sequence execution until the message is acknowledged or until a timer expires. If the computer is set up with network 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.
 - 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 reduced remanence.

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 close to the set point, and will then slow the rate 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.

- **Standby** – Places the instrument in a ready-to-use state. The field is set to zero, the magnet controller is turned off, the chamber temperature is set to 300K, and the chamber is sealed.
- **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, 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 77 K 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 instrument standby.)

3.5.5.2 MEASUREMENT COMMANDS

Log Data – Records specified diagnostic data to a data file at a specified rate. No size limit is imposed on the data file generated, so the data file can get extremely large and difficult (or slow) to process. See Appendix B for more information on logging diagnostic data.

- **Sigma Log Data** – Records specified diagnostic data to a data file at a specified rate. This command is similar to the Log Data command, except that it allows you to record statistics such as a running average and standard deviation for each data item, in each data record. It may generate large data files with large header sections. See Appendix B for more information on logging diagnostic data.
- **Measurement Option Commands** – If a measurement option is active, an additional branch will be present populated with sequence measurement commands. See the appropriate option manual for instructions on how to use these commands.

3.6 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,” by selecting “File > Open > Datafile,” or on the control center click “View” to open the current data file.

3.6.1 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...”
- The axes may be assigned any label for which data exists in the data file.
- Up to four vertical axes may be displayed on separate plots in the same graph window, but all plots in the graph window must share the same horizontal (x) axis.
- When auto-scaling is selected for all axes, the plot axes will be rescaled each time new data is written to the data file so that all data is displayed. This helps optimize data viewing as data is being collected.
- Quickly auto-scale the axes by right clicking in the graph window and selecting “Auto Scale All Plots.”
- Use the “>>” button to add custom axis labels and to change the axis scaling multiplier by factors of 10.
- Use the “Filter...” button to plot only records with data that falls within a specified range. For example, specify a range for moment standard error to hide data with a large standard error (i.e. noisy data.).
- The “OK” button applies the changes to the graph window and closes the Data Selection dialog. The “Apply” button applies the changes to the graph window without closing the dialog. The “Cancel” button closes the Data Selection dialog without making any changes to the graph window.

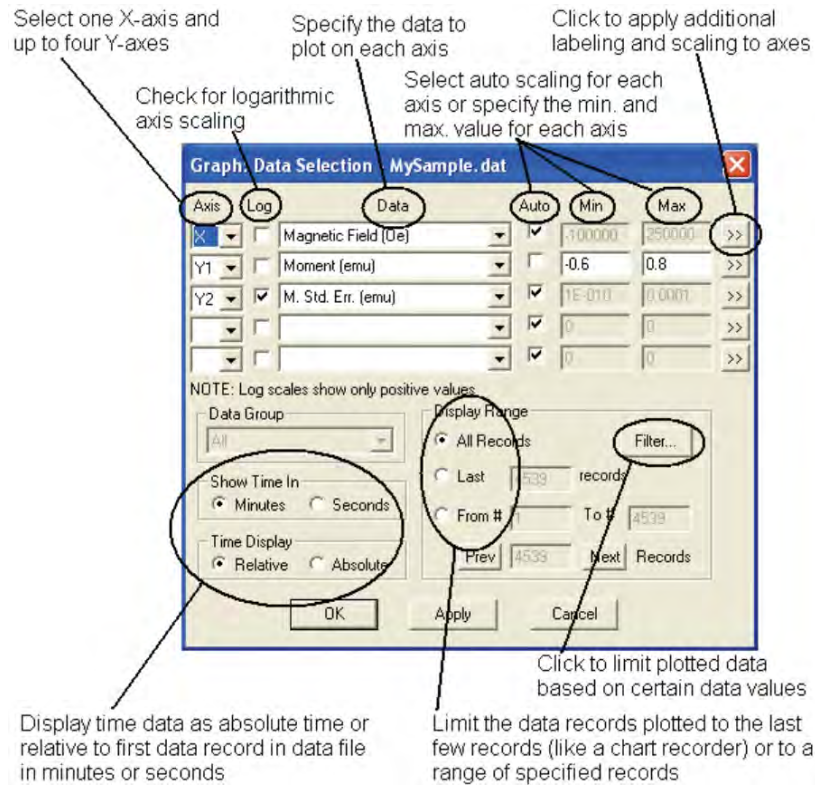


Figure 3-18. Graph Data Selection Dialog

3.6.2 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 directions using the “>>” button.
- Check “Apply to All Plots” to apply the appearance settings to all plots in the graph window.

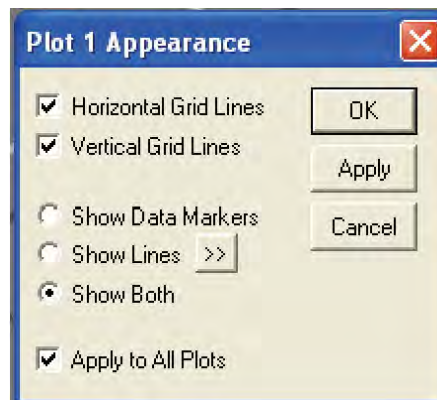


Figure 3-19. Plot Appearance Dialog

3.6.3 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. The settings are specific to files a given type, so applying a template file from a Log Data file to a VSM Data File will yield unexpected results.

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 defaults that are specified in the data file when the file is created. For measurement options, the default setting usually displays the data you are most likely to look at (e.g. Sample Heat Capacity vs. Temperature for the Heat Capacity Option.)

3.6.4 Exporting Data Files

Many spreadsheet programs can read the standard .dat files VersaLab 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, do the following:

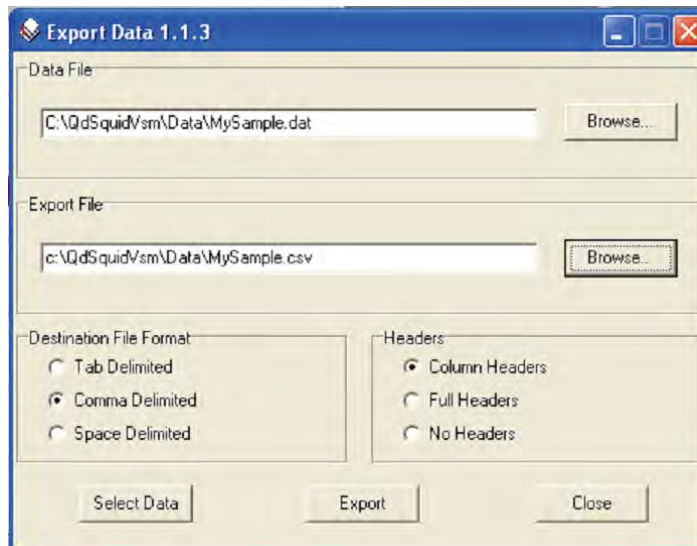


Figure 3-20. Exporting Data

- Select a graph view of the data file you want to export.
- Select the menu item “File > Export...”
- Make sure “Data File” specifies the file you want to export. You can browse to a different data file if desired.
- Designate the path and file name of the exported file to write under “Export File.”
- Under “Destination File Format,” choose which character will separate data items in the exported 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 of 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 MultiVu data 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 exported file. You will see a confirmation dialog when the operation is complete.

System Operation

4.1 Overview

This chapter describes the operation of the VersaLab system. You should read Chapter 2: Hardware and Chapter 3: Software before performing any tasks described in this chapter. Chapter 1: Common Tasks describes several common system operation tasks that are not described in this chapter.

Section 4.2 describes how to set up VersaLab, including connecting the helium compressor. Section 4.3 gives instructions on cooling down the system. The remaining section describes the utility wizards, which guide the user through warming and cooling the system, resetting the magnet, and cryopump regeneration.

4.2 System Set Up

Quantum Design service personnel will typically perform system set up. It is possible for you to set up the equipment yourself, so instructions are included here. Please read this section and contact Quantum Design service before attempting to set up the instrument.

Setting up the VersaLab system involves four steps, detailed in the sections below:

- Connecting the compressor
- Connecting the vent gas
- Connecting power
- Setting up the computer

Before performing these tasks, you should determine where to place the equipment. VersaLab may be placed with either side against a wall, if desired. The rear of VersaLab may be close to a wall, but enough gap is needed to observe the minimum bend radius of the compressor lines, 30 cm (12 inches). The compressor must be placed such that it receives adequate ventilation. Air enters through the vents in the side of the compressor, and exits through a vent in the rear. Be sure there is at least 15 cm (6 inches) of clear space around all sides of the compressor.

The compressor is on casters, so it can simply be wheeled to the desired location. The compressor is quite top-heavy so take care not to tip it.

VersaLab may be moved in one of two ways:

1. One person may roll VersaLab to the desired location using the wheels on the back of the assembly. To do this, place one foot on the wheel axle and pull back on the round stainless steel handle at the rear of VersaLab as shown in the figure. Then simply roll the instrument to the desired location. Take care not to suddenly lower the instrument, as doing so could cause damage.
2. Two people may simply lift VersaLab and carry it. To do this, two people stand, one on either side of VersaLab, and pick up the instrument by the rectangular cross-section stainless steel bar. Carry it to the desired location and carefully set it down.

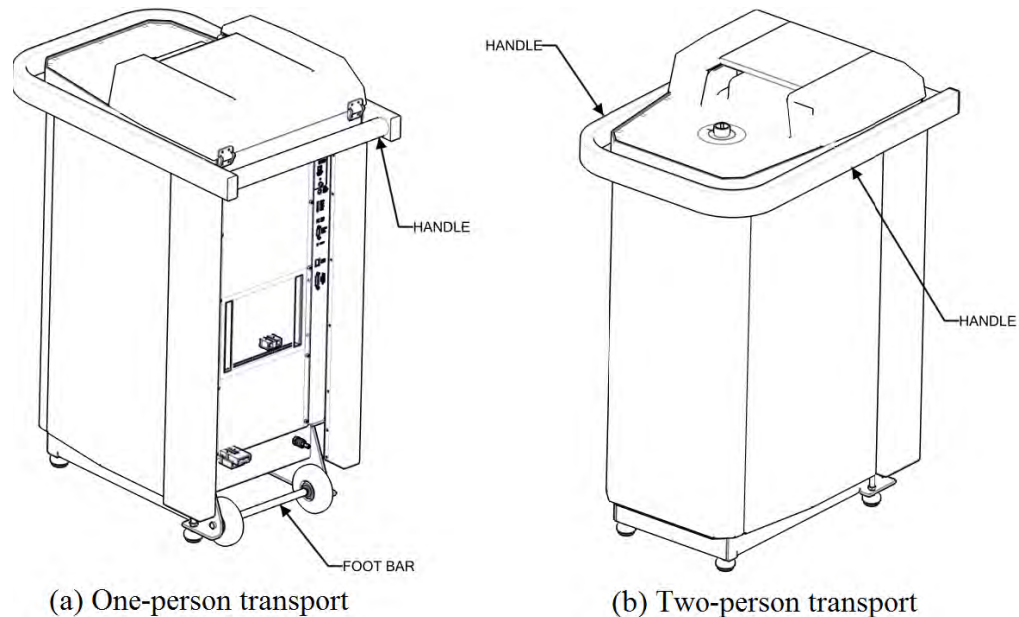



Figure 4-1. How to move VersaLab with one person (a) or two people (b).

	WARNING!
<p>Be careful when moving VersaLab. Do not drop the instrument, because doing so could cause damage. Do not attempt to lift the instrument by yourself, because doing so could cause injury. Take care not to damage the high-pressure gas lines when moving the instrument.</p>	


4.2.1 Connecting the Compressor

Once you have determined where to place the VersaLab instrument and the compressor, connect the high-pressure helium gas lines and the cold head motor drive cable to the compressor. VersaLab is delivered with the gas lines and motor drive cable already connected to the instrument, so you need only connect them at the compressor. Then set switches on the compressor so that it is configured for your power and is ready to start.

WARNING!

Follow these precautions to avoid injury and damage during this procedure:

- Wear safety glasses when connecting or disconnecting high pressure gas lines.
- Always use back-up wrenches to avoid damaging the compressor or venting gas.
- Do not bend the compressor lines with a radius less than 30 cm (12 inches).
- Keep caps on the gas fittings until you are ready to make connections in order to avoid getting dirt in the fittings.
- Take care not to cross-thread the fittings. The fittings have fine pitch threads that are easy to cross thread.
- Always connect the RETURN fitting first. Do not connect the SUPPLY fitting first, as doing so could cause contaminants to enter the cold head.



First, connect the RETURN gas line to the RETURN gas fitting on the compressor. To do this, finger-tighten the female fitting (on the gas line) onto the male fitting (on the compressor). Then use three wrenches to tighten the fitting. One 5/8 inch wrench is used on the gas line to prevent un-mating an O-ring connection and venting gas. One 3/4 inch wrench is used to prevent the male fitting on the compressor from turning. Finally, another 3/4 inch wrench is used to tighten the female fitting on the gas line onto the compressor. Tighten the fitting until it stops. High torque is not required in order to obtain a seal.

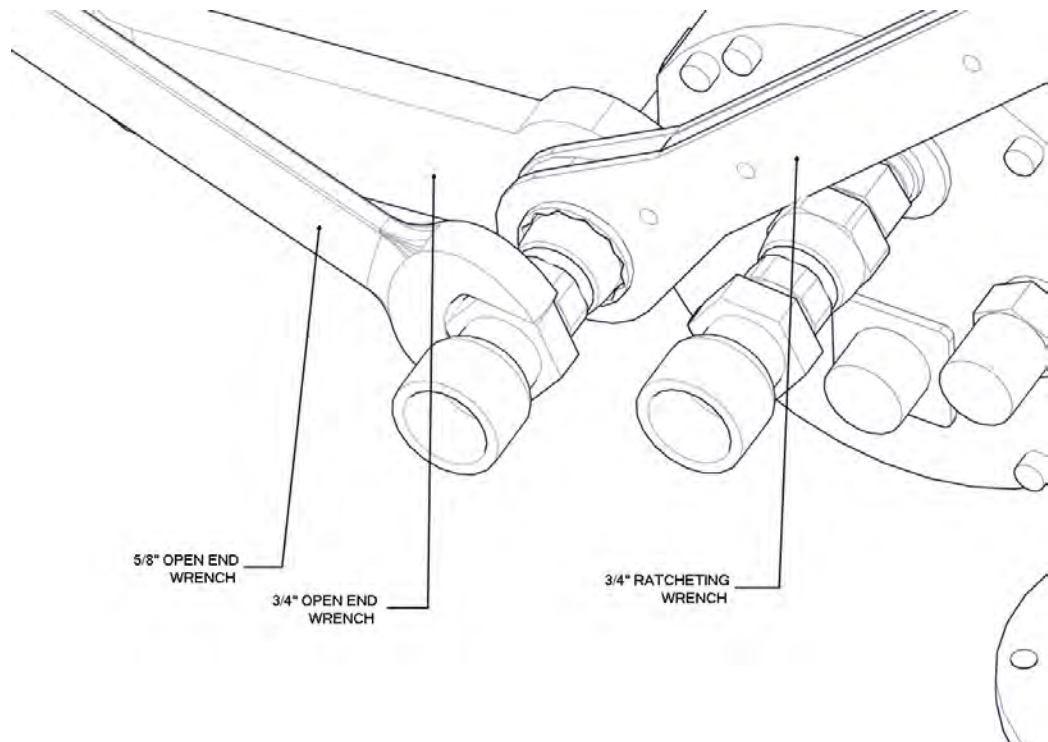


Figure 4-2. Wrench placement when attaching high pressure lines.

Second, connect the SUPPLY gas line to the SUPPLY gas fitting on the compressor. Perform this procedure as you did for the RETURN line.

Finally, connect the cold head drive military-style connector on the compressor.

4.2.2 Connecting Vent Gas

There is no dewar gas available for venting the sample chamber in VersaLab, so you must supply a gas bottle. There are two common choices for vent gas.

- Helium. For most applications, you use helium gas. If you plan to perform measurements below room temperature, you should generally use helium gas. Unlike other gases, helium will not condense on samples or bearing surfaces. Quantum Design supplies the sample chamber pressure gauge calibrated for helium.
- Nitrogen. If you will be performing measurements only at room temperature, you can use nitrogen gas. Do not use nitrogen gas for any low temperature measurements that are sensitive to condensed or frozen gases such as Heat Capacity (where the gas will condense on the sample and be measured) and Vibrating Sample Magnetometer (where the gas will condense on a bearing surface and cause noise). The sample chamber pressure gauge requires recalibration for use with nitrogen. Contact Quantum Design service for recalibration instructions.

VersaLab is supplied with a length of polyethylene tubing and a gas regulator in order to connect to a gas bottle. Note that the regulator is supplied with a standard CGA-580 fitting. You are responsible for providing any necessary adapters if your gas bottle requires a different fitting.

After securing the gas bottle, attach the regulator to it using the CGA fitting. Determine the appropriate length of polyethylene tubing to connect VersaLab to the regulator, and cut the tubing to length. Be sure to cut the end of the tubing perpendicular to its axis in order to ensure a good seal. Next, place the supplied compression nut on the tubing along with the plastic ferrule set. Finger-tighten the compression fitting onto the mating fitting on the regulator, then turn another 1/2 turn. Do not tighten excessively.

Next, set the regulator. Loosen the pressure adjustment knob on the regulator to close the regulator. Open the gas valve on the bottle. Slowly tighten the pressure adjustment knob to obtain a pressure between 1 and 2 psi. Close the needle valve on the regulator, then open it 1/2 turn. These settings ensure adequate vent gas supply without wasting excessive gas during a Vent Continuous operation.

You leave the gas bottle open at all times so that vent gas is available to VersaLab.

Finally, leak check the compression fitting that you connected at the regulator. A leak at that fitting may empty a gas bottle in a short period. Use soapy water and look for bubbles in order to leak test this connection.

4.2.3 Connecting the Power

All electronics in the VersaLab cabinet and the helium compressor are powered from a single wall outlet. This configuration avoids ground loops and potential safety hazards associated with the equipment. The instrument is rated for 200-240 VAC 50/60 Hz.

To power the system:

1. Check that the power switch is set to off.
2. Connect the supplied power cord to the power entry on VersaLab.
3. Slide the power entry clamp down the cable to the power entry. Attach it to VersaLab using the two screws provided.
4. Connect the compressor to VersaLab using the power outlet at the rear of the instrument.



WARNING!

CAUTION: The instrument must be plugged into a single phase IEC 60309 outlet (16 A/250 V) fused at 16 A. See Appendix C.2 for details. Always power the system down before unplugging the power cord.



On the system side, the power cord is secured to the instrument by a clamp. Do not tamper with this setup unless specifically instructed by qualified Quantum Design service personnel. Risk for electrical shock hazard.



The main power switch is a resettable breaker rated at 16 A.

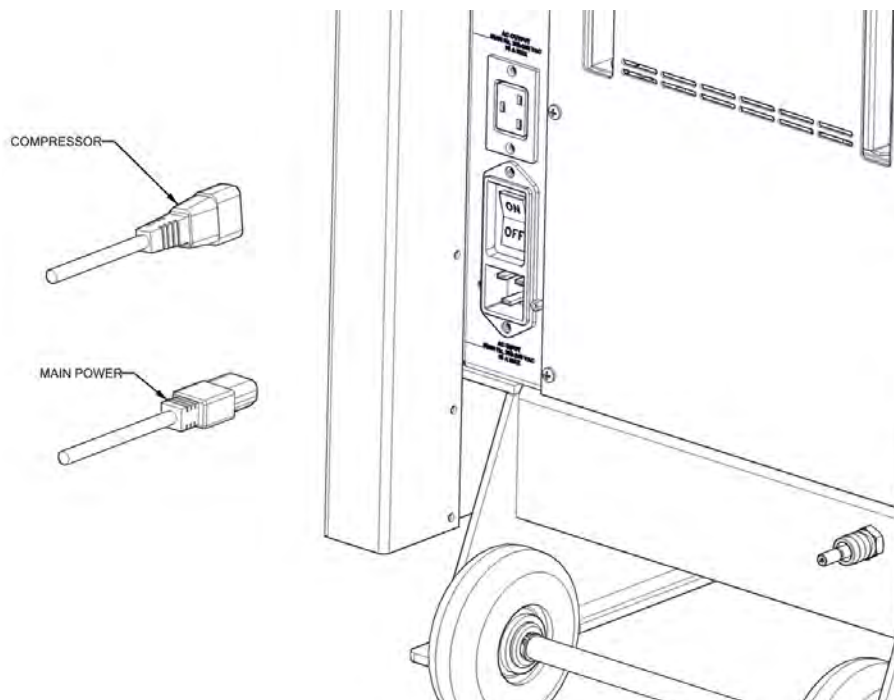



Figure 4-3. VersaLab's Power Connections

4.2.4 Setting Up the Computer

The computer may be powered from any voltage in the range 100-250 V and does not need to be connected to the same power source as VersaLab and the compressor. Some computers require you to change a setting at the power input to match your laboratory's power source.

	WARNING!
	Check the voltage setting of the computer power input before connecting power to the computer. Failure to do so may destroy the computer's power supply.

After verifying that the computer voltage setting is correct, power it from any outlet that supplies the appropriate voltage.

To connect the computer to VersaLab, simply connect the supplied USB cable to the port labeled "JQG-1 USB-CAN" on the rear of VersaLab and to an available USB port on the computer.

4.2.5 Option Setup

Most measurements that you perform with VersaLab require the use of measurement options. Measurement options are described in separate manuals. Consult those manuals for setup and operation instructions. Use of some measurement options may require optional CAN modules to be installed in the Versalab module bay. Refer to Section 3.2.5 for procedures and warnings regarding CAN modules. Instructions regarding installing option software may also be found in Chapter 3.

4.3 System Cool-down

In order to cool down the system, use the wizard "Cool-down Wizard." To access the wizard, select the MultiVu menu item "Utilities > Wizards > Cool-down Wizard." The wizard will first perform a series of system checks. Once the system checks are complete, follow the on screen instructions as the wizard walks you through the following steps to cool the system down.

Note: Depending on your compressor type, the wizard may automatically disable/enable remote control of the compressor when necessary. If your compressor type does not support these automated functions, the wizard will instruct you when and how to manually perform these compressor functions.

1. Disconnect the vent gas using the quick disconnect and seal the sample chamber in order to allow VersaLab to purge the cryostat case with air and remove any helium. The

wizard will not proceed until these steps have been checked off by the user.

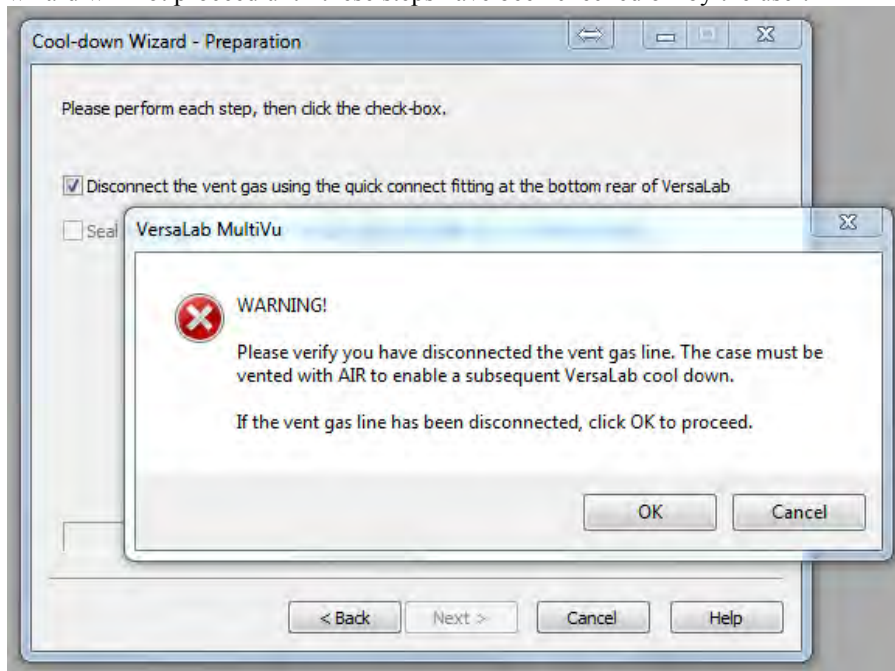


Figure: 4-4 Vent gas disconnect warning

Warning! If the vent gas is not disconnected at this time the vacuum case will be flooded with helium. Even small amounts of helium gas in the vacuum case will act as a thermal short and will prevent the VersaLab from cooling down properly.

2. Wait while VersaLab purges and pumps out the vacuum case and regenerates the cryopump. This step will run unattended.

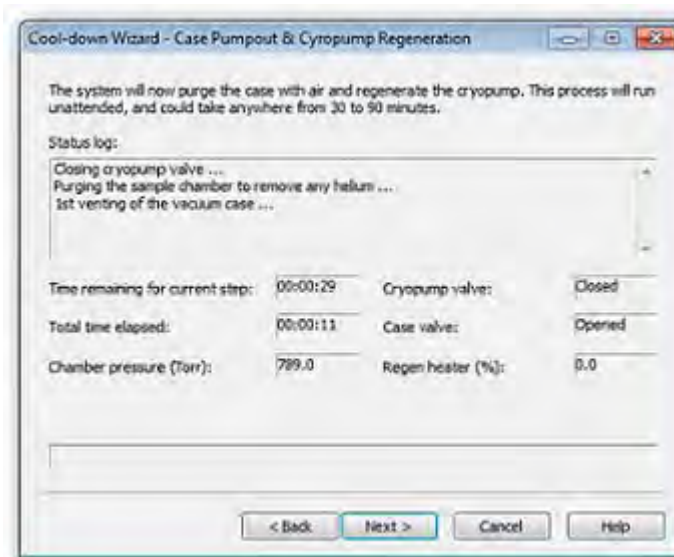


Figure 4-5. Cool-down wizard pumpout and regeneration

3. Follow the procedures to start the cryocooler in order to cool the system down.

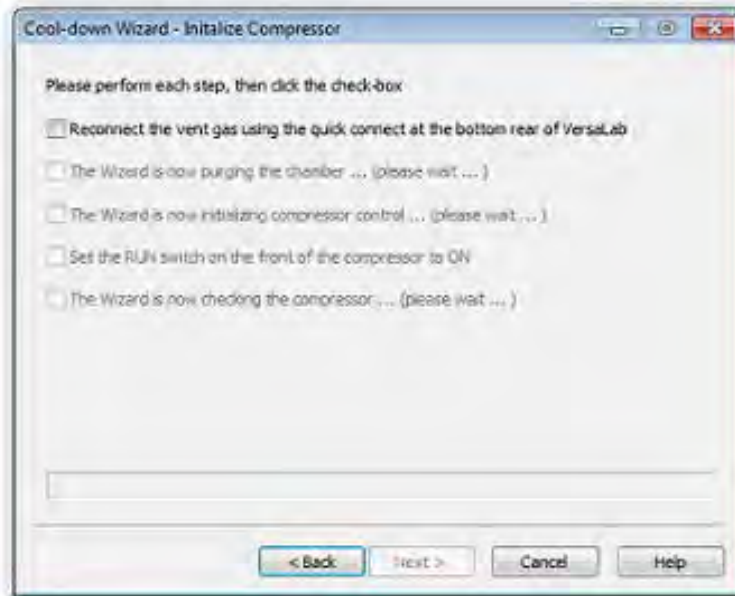


Figure 4-6. Cool-down wizard compressor initialization

4. Progress of the cool-down will then be monitored through the wizard. Temperature and field control will resume once the cool-down is complete.

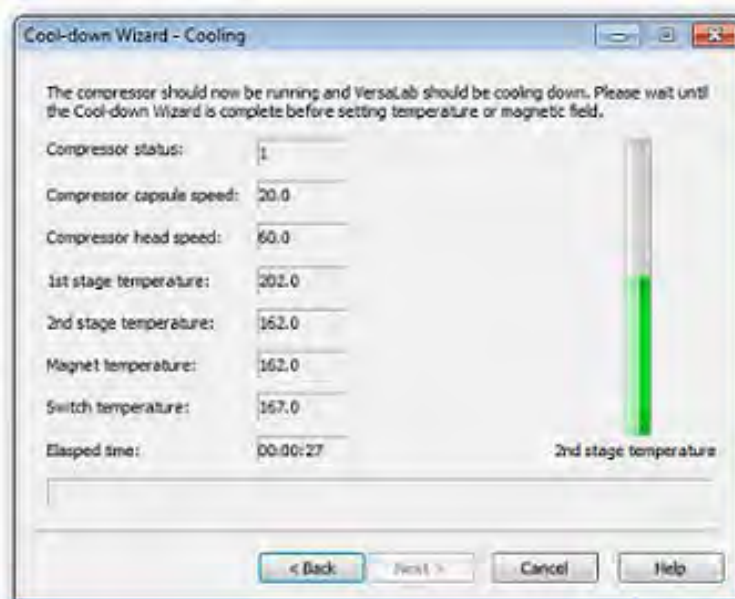


Figure 4-7. Cooling progress

4.4 Other Utility Wizards

In addition to the Cool-down Wizard, there are several other wizards which guide the user through certain VersaLab functions. The “Utilities” menu includes wizards for system warm-up, leak-checking the VersaLab, resetting the magnet, and regenerating the cryopump. The general steps carried out in each wizard are given in the following sections.

4.4.1 Warm-up Wizard

If you do not plan to use VersaLab for more than one week, then you may wish to shut it off and allow it to warm up. Quantum Design recommends that you leave the system cold if it will be idle for shorter periods due to extra wear on the cold head during a cool down.

In order to warm the system up, run the warm up wizard using the MultiVu menu item “Utilities > Wizards > Warm-up Wizard.” Follow the on-screen instructions as the wizard walks you through the following steps to warm the system up.

1. Disconnect the vent gas using the quick disconnect in order to allow VersaLab to vent the cryostat case with air (not helium).

Warning! If the vent gas is not disconnected at this time the vacuum case will be flooded with helium. Even small amounts of helium gas in the vacuum case will act as a thermal short and will prevent the VersaLab from cooling down properly.

2. Warm the sample chamber to room temperature.
3. Turn OFF the cryocooler.
4. Vent the case partially with air.
5. Pump out the chamber to avoid excessive accumulation of contaminants in the chamber. Leak Check Wizard

The leak check wizard should be used if you suspect that there is a leak into the VersaLab cryostat. Details of the Leak Check Wizard can be found in Section A.7.

4.4.2 Leak Check Wizard

The leak check wizard should be used if you suspect that there is a leak into the VersaLab cryostat. Details of the Leak Check Wizard can be found in Section A.7.

4.4.3 Reset Magnet Wizard

The VersaLab magnet has a typical remanence < 4 Oe. If you wish to achieve a lower field, reset the magnet. The VersaLab magnet reset operation involves simply turning off the cryocooler and

waiting until the magnet warms above its superconducting critical temperature. The cryocooler is then turned back on to allow the magnet to cool back down.

To reset the magnet, use the MultiVu menu item “Utilities > Wizards > Reset Magnet Wizard.” Follow the on screen instructions as the wizard guides you through the following steps:

1. The wizard will warm the sample space, discharge the magnet, and purge the chamber. The wizard then puts the system in standby.

The cryocooler will be turned OFF (or the wizard will instruct you to do so).

2. Wait for the magnet to warm up as indicated by the wizard.
3. *Note: If your compressor must be turned on manually, be sure to stay near the instrument during this time, because it will take longer to cool back down if you wait too long.* The cryocooler will be turned ON (or the wizard will prompt you to turn the compressor on manually).
4. Wait for the system to cool back down as indicated by the wizard. Field control will be disabled until the magnet has cooled back down to the necessary temperature.

4.4.4 Cryopump Regeneration Wizard

As discussed in Section 2.5.4, contaminants and helium gradually build up in the cryopump. The cryopump is always regenerated before cooling down the system in order to remove the contaminants. However, if the system has been cold for a long time and you use the cryopump frequently, you may find that the cryopump no longer achieves high vacuum. In that case, regenerate the cryopump using the MultiVu menu item “Utilities > Wizards > Regenerate Cryopump Wizard.” The wizard asks you to decide whether to perform a quick regeneration or a complete regeneration. Perform a quick regeneration if you suspect that the cryopump is simply saturated with helium. Perform a complete regeneration if you suspect the cryopump contains large quantities of contaminant gases- a situation that is more likely if you frequently pump on parts with large surface area, such as the VSM linear motor during oven operation. The complete regeneration warms the system considerably, and so requires about 3 hours to complete. If you are unsure whether a complete regeneration is required, try a quick regeneration first.

After you seal the sample chamber KF flange, the wizard performs the regeneration with no user intervention. However, once the regeneration is complete, the system will be left in standby. If you wish to use the system after running the wizard, the cryocooler will need to be activated by double-clicking on the cryocooler panel in the MultiVu status bar (see section 3.3.5) and selecting “Activate System.”

Note: For the Full Cryopump Regeneration, the wizard will not declare the regeneration as complete until the 2nd stage temperature has re-cooled and maintained temperature below 5K for a period of 20 minutes.

Maintenance and Servicing

A.1 Overview

This appendix describes the most common maintenance procedures needed to keep VersaLab running properly. Also consult measurement option manuals for maintenance of option hardware. Be sure to contact Quantum Design service if you ever have questions about system maintenance.

A.2 Sample Puck Adjustment

You use the puck adjustment tool on the sample puck after you have inserted the puck into the sample chamber approximately 10 times or whenever the puck fits loosely into the bottom of the sample chamber. Figure A-1 illustrates the puck adjustment tool.

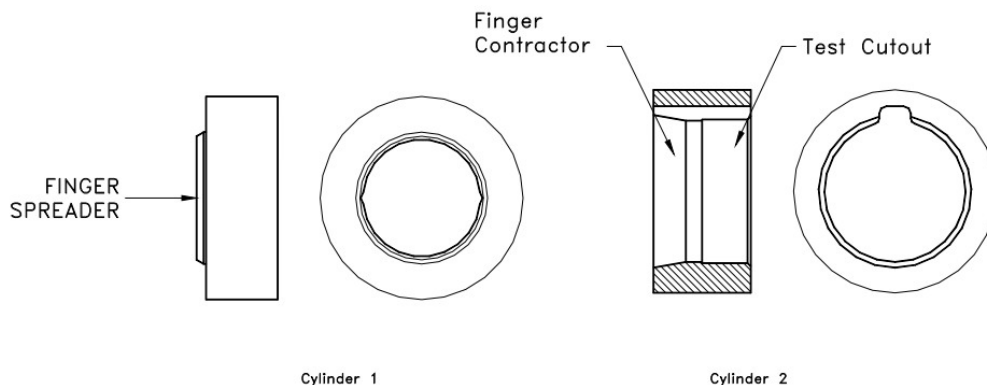


Figure A-1. Puck Adjustment Tool

Complete the following steps to use the puck adjustment tool:

1. Place the puck on the finger spreader. Refer to Figure A-1. If there are two small screw heads on the bottom of the puck, such as on a heat capacity puck, be sure to align them with the cut outs on the spreader.
2. Press the puck downward and continue pressing until all the chuck fingers touch the base of the finger spreader. When all the fingers touch the base of the spreader, the spreader evenly applies radial force to the fingers, pushing them outward and slightly beyond their optimal location.

3. Remove the puck from the finger spreader.
4. Place the puck inside the finger contractor. Refer to Figure A-1.
5. Press straight down on the puck and continue pressing until you press the puck completely into the finger contractor. When the entire puck is in the contractor, the contractor evenly applies force to the outside of the fingers, pushing them inward. The contractor pushes the fingers—regardless of external wear or variations on the puck—so that the fingers obtain their optimal location.
6. Remove the puck from the finger contractor.
7. Place the puck inside the test cutout. Refer to Figure A-1. Verify that the puck fits easily but snugly inside the test cutout. Do not insert the puck into the sample chamber if it does not fit into the test cutout, doing so could cause damage to the sample chamber.

A.3 Inspecting and Maintaining O-rings

O-rings require regular inspection and lubrication. The only O-ring that you regularly encounter in VersaLab is the one on the sample chamber KF flange. Be sure to maintain this O-ring as described below. In addition, if you service any other parts of the system, be sure to inspect and lubricate O-rings you encounter during servicing as well.

Whenever you see an O-ring, take the time to visually inspect it. If it appears dirty, clean it with a lint-free cloth. Replace O-rings that are cracked. If the O-ring is dry, apply a small amount of silicone vacuum grease to it. Apply only a thin film of grease to the O-ring. Do not apply so much grease that more than a film is visible.

Also inspect the metal surfaces that mate with the O-ring, and clean them if necessary. Take care not to use sharp instruments on O-rings or the mating surfaces as it could result in damage.

Keeping O-ring seals in good working order prevents leaks that could cause malfunction or damage to the instrument. Leaks at the sample chamber KF flange can cause the formation of an ice plug in the sample chamber, which results in dangerous and damaging pressure during warming. Be sure to maintain this often encountered O-ring.

A.4 Cryocooler Maintenance



WARNING!

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse or improper operation of the system. This product is NOT operator-serviceable. Please contact your Quantum Design customer service representative for any service issues.

The cryocooler that cools VersaLab requires maintenance to operate properly. Quantum Design personnel should perform this maintenance unless you discuss performing the maintenance yourself and receive separate instructions describing the maintenance procedures. Do not attempt to perform any cryocooler maintenance without proper instruction from Quantum Design.

There are two cryocooler maintenance items: the cold head rebuild and replacement of the compressor adsorber. For both, you need to consider how many hours the cryocooler has been operating. You read this number on the display on the front of the compressor.

A.4.1 Cold Head Rebuild

After operating for 10,000 hours, the cold head performance may noticeably degrade due to wearing of its seals. The time for noticeable performance degradation may be considerably greater than 10,000 hours. It is not necessary to perform the maintenance until the performance degrades to the point that your VersaLab system is no longer functional. No damage is done to the cold head if you continue to use it as the performance degrades. Because the rebuild process involves cost and down time, Quantum Design recommends that you wait until you experience performance degradation before having the cold head rebuilt.

In order to check for performance degradation, use Log Data or the Background Log (sections B-2 and B-3) to determine the first stage, second stage, and magnet temperatures. Read the temperatures when the system has been in standby for several hours. When the cryocooler is new, the temperatures are typically: first stage < 35 K, second stage < 3 K, and magnet < 3.5 K. You will start to notice system performance issues when the temperatures are: first stage > 40 K, second stage > 4 K, magnet > 5 K. Your first indication of cold head wear might be frequent magnet quenches due to a warm magnet. If you are unsure about the status of your cold head, send the Background Log file to Quantum Design service for evaluation.

The cold head rebuild involves warming up the system, disconnecting the cold head from the compressor, depressurizing the cold head, removing the displacer assembly, and replacing it with a rebuild displacer assembly. The cold head is then flushed with clean high purity helium.

A.4.2 Compressor Adsorber Replacement

The compressor adsorber removes oil vapor from the compressed helium gas before it is supplied to the cold head. Any oil in the helium would freeze in the cold head, quickly damaging its seals. Therefore, it is very important to replace the adsorber as recommended by the manufacturer. It is recommended that the adsorber be replaced every 30,000 operating hours.



WARNING!

Make sure that you replace the adsorber every 30,000 operating hours or sooner. Failure to do so may result in premature wear of the cold head, requiring costly rebuild.

Replacing the compressor adsorber involves warming up the system, disconnecting power to the compressor, removing panels to access the adsorber, and replacing the adsorber. Self-sealing fittings are used, so the system need not be purged after this operation. Addition of helium gas may be necessary.

A.4.3 High Pressure Helium Gas Charging

The Helium Compressor comes pre-charged with High Purity Helium, yet it may be necessary for the user to recharge the unit in the case of a vent.

Tools Needed

- High Purity Helium Gas ($\geq 99.999\%$)
- Compressor Charging Kit (4601-931) which includes:
 - o Helium Compressor Charging Hose (4601-920)
 - o Helium Compressor Gas Regulator Assembly (4601-930)
 - o Vent (RF-04) Fitting Fixture (4601-916)
 - o “SNOOP” Liquid Leak Detector Fluid (HM751)

A.4.3.1 PROCEDURE: PREPARE THE CHARGING KIT

Prior to charging the Compressor Unit the Charging kit needs to be flushed with High Purity helium Gas:

1. Mount the Helium Compressor Gas Regulator (4601-930) to the Gas Cylinder: **DO NOT OPEN THE GAS CYLINDER AT THIS TIME!**
2. Ensure that the Valve of the Gas Regulator is open.
3. Connect the Charging Hose (4601-920) to the Regulator.
4. Screw onto the open end of the charging hose the Vent Fitting Fixture (4601-916).
5. Slowly open the Gas Cylinder. Helium Gas is now blowing out of the Vent Fixture.
6. After 15-20 seconds, unscrew the Vent Fixture from the Charging Hose while the Helium Gas is blowing. The Charging kit is now ready for use.

A.4.3.2 CHARGE HELIUM COMPRESSOR

1. Connect the Purged Charging Hose to the Helium Filling port of the Compressor

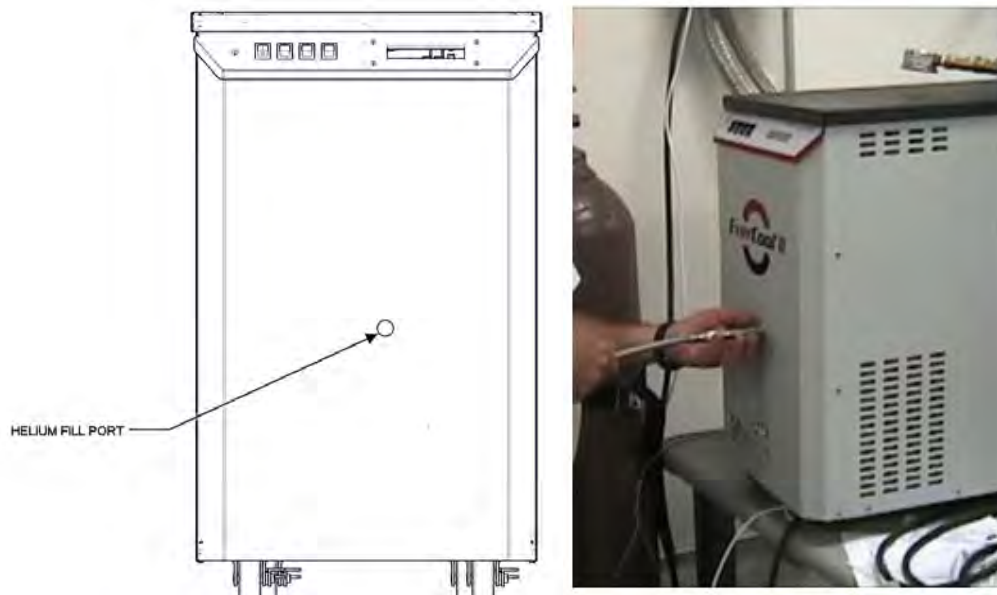


Figure A-2. Connect Charge hose to the Helium Fill Port

2. Quantum Design Compressors: Turn on the Main Power Switch of the Compressor Unit.
3. Quantum Design Compressors: Scroll through the Compressor Display by toggling the Display switch to display the system pressures.
4. Sumitomo Compressors: Read the pressure from the analog gauge on the front of the compressor.
5. Slowly increase the Pressure on the Regulator to the value specified in the table below.
6. Scroll through the Compressor Display and check that the Static Pressure is within 1.72 to 1.78 MPa.

If it is necessary to purge the system, be sure to attach the purge line to the supply line of the helium compressor. Attaching the purge line to the front fill port or the return line would cause helium to flow in an unintended direction, which could cause damage to the compressor.

A.5 Cosmetic Panels, Removing and Installing

The cosmetic panels on the left and right side of the system can be removed to gain access to the main controller board (left panel as facing the system) and main power supply (right panel as facing the system).

To remove panels pull the back portion of the panel away from the system. This will disengage the ball studs on the back of the panel. Next, slide the panel towards the back of the system freeing the panel from the grommets that secure it to the front panel.

To reattach the panels, reverse the process. Slide the front posts of the panel into the grommets of the front panel, and engage the ball studs in the back.

A.6 Replacing Fuses and Resetting Breakers

The system has three user accessible fuses as well as a breaker on the power input. In case you encounter situations where a fuse blows or a breaker jumps, contact your Quantum Design service representative to investigate the root cause of the problem.

A.6.1 Power Input Module

The main power entry located on the rear of VersaLab has a 16 A breaker-switch. In the event of over current, the breaker will switch to the off position. It may be switched back to the on position like a normal rocker switch.

A.6.2 Controller Board



The controller board utilizes three fuses. To access these fuses, first disconnect the system from the wall power outlet. Then remove the left cosmetic panel as described Section A-5. The three fuses are labeled on the PC board, and should be replaced with fuses of the values indicated here. Do not use fuse values other than these:

- Fuse F1 is a 0.25 A, 20 mm delay type fuse.
- Fuses F2 and F3 are both 2 A, 20 mm delay type fuses.

A.7 Leak Checking the System

If VersaLab does not cool down properly, or the diaphragm pump is unable to pump down the cryostat before cooling down the system, then there may be a leak into the cryostat. If you have a helium leak detector available, you can use it to look for leaks. You will need to supply the appropriate fitting to connect your leak detector to the KF-40 flange on the VersaLab sample chamber.



WARNING!

If you choose to leak check VersaLab, be sure that your leak detector will not back stream any oil into the system. If oil enters the cryostat, it is very difficult to remove and may prevent proper operation.

Before leak checking the system, VersaLab must be fully warmed up. In order to check for a leak, use the Leak Check System wizard (Utilities > Wizards > Leak Check System). The wizard guides you through the following process:

1. Connect the leak detector to the VersaLab sample chamber.
2. After you indicate that you have made the connection, the wizard connects the cryostat to the sample chamber.
3. Start the leak detector pumping on VersaLab when the wizard indicates that it is time to do so.
4. Wait for the background to decay to an acceptable value. Typically, a background of less than 10^{-7} scc/s is adequate.
5. Leak check the system.
6. The wizard closes the connection between the cryostat and sample chamber when you indicate that you are done leak checking.
7. Stop the leak detector when the wizard indicates that it is time to do so. Be sure that you stop the leak detector at this step, because otherwise the system will vent your running leak detector at the next step.
8. Disconnect the leak detector after the system vents the sample chamber.

Diagnostic Software Operations

B.1 Overview

This appendix describes diagnostic software operations that help you troubleshoot problems with your VersaLab instrument. Often you will use these features at the request of Quantum Design service personnel when you report a problem with the instrument. Data that you collect using these procedures can be sent to Quantum Design service to speed the diagnosis of your problem.

B.2 Log 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 where the data will be saved. You may specify a new file name or an existing log data file. 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.

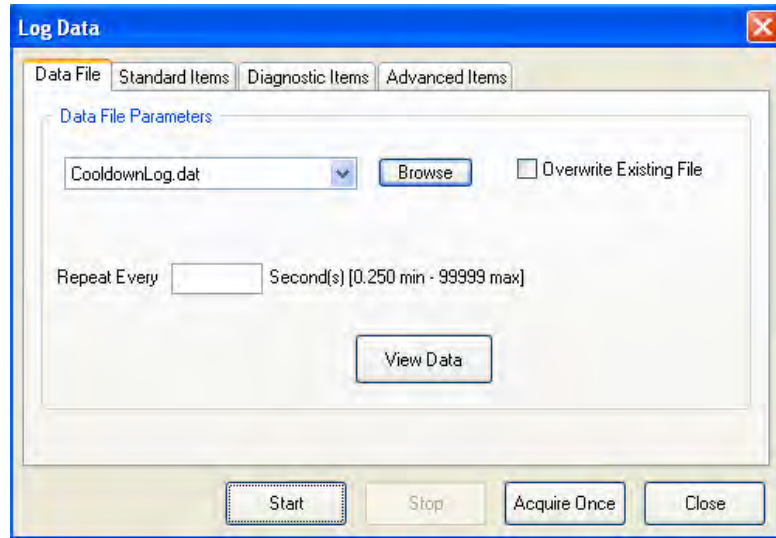


Figure B-1. Data Logging Interface

The menu command “Utilities > Sigma Log Data...” contains very similar capabilities to the log data command, except that you may log the statistics (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 following three sections describe the data available on the tabs of both the Log Data and Sigma Log Data dialogs.

B.2.1 Standard Items

The standard items tab contains information that is mostly available in the MultiVu status bar. The status of the chamber gas, temperature, magnetic field, and cryocooler are recorded in the file as integers. The log data window displays the meaning of these status codes in real time, but this text is not included in the log data files. (Use “Utilities > Status Calculator...” to decode status codes. The “Query” button will decode the current state of the instrument.)

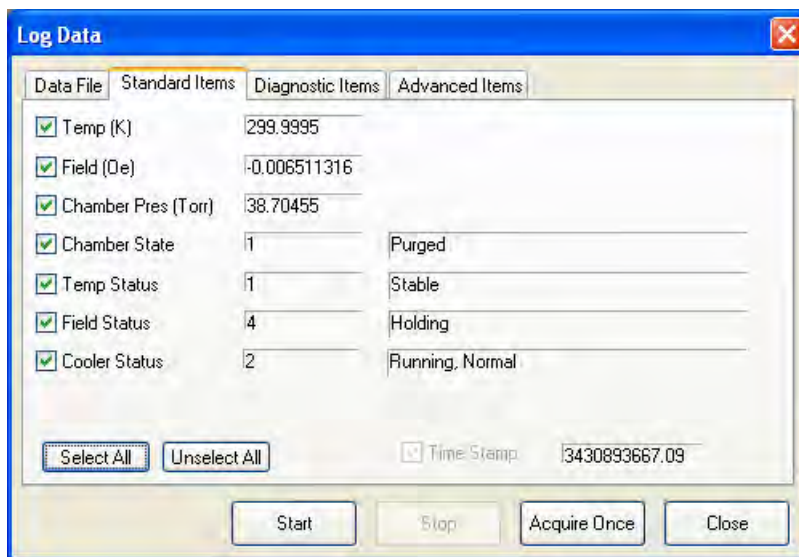


Figure B-2. Standard Items Tab

B.2.2 Diagnostic Items

The diagnostic items tab contains detailed information about the instrument state that is useful for troubleshooting. Problems with temperature control, magnetic field control, and the cryogen-free cooling system can all be diagnosed using data in this tab.

B.2.3 Advanced Items

Advanced Items are all configurable data items. A large amount of raw instrument data is available for logging when required. Data can be logged from any CAN node on the bus. Configuration of these data items is accomplished with a map (*.qmap) file which is usually supplied by a Quantum Design representative. The map file is loaded using the "Select Map File" button and the resulting browse dialog.

B.3 Background Log

Any time MultiVu is running, it records diagnostic data to a background log file. The file (C:\QDVersaLab\MultiVu\BRLog.dat) is a normal MultiVu data file that can be viewed like any other data file. The background log file is limited in size to 20 MB. When this limit is reached, the first half of the file is removed so that the most recent data are always available.

In case a problem occurs when you are not running Log Data, the problem might be diagnosed using the background log. Quantum Design personnel may ask you for this file if you report a problem with your instrument.

B.4 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. When this limit is reached, the first half of the event log is removed so that the most recent data are always available in a file of reasonable size. The file 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 VersaLab, 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), MultiVu displays Event Notification dialog with the three most recent events. This ensures that you don't miss potentially significant error messages.

System Requirements, Pin-out Diagrams, and Interconnect Diagrams

C.1 Overview

This appendix contains system requirements and information that is useful to understand how the various components of the system are connected. Pin out diagrams allow you to determine where in a connector various signals are located. Diagrams show how the signals, power, and gas flow are routed in VersaLab.

C.2 System Requirements and Operating Conditions

The following install requirements are to be provided as minimum supply requirements for proper operation of the VersaLab. The installation of the VersaLab and any associated equipment shall follow local electrical and building codes. For specific requirements for the compressor, please refer to the information provided by the equipment supplier. Some of the installation must be performed by a qualified Quantum Design representative and you will need to schedule installation service.

C.2.1 Power Ratings



Voltage: 200-240 VAC, 50-60 Hz, single phase

Current: typical operation 10 A



Mains Fusing: 16 A breaker at main power inlet

¹Product Tested at +/- 10 % of stated range.

C.2.2 Laboratory Ambient Conditions

Location for use:	Indoor only
Operating ambient temperature:	10-35 °C (50-95 °F)
Operating ambient relative humidity:	10-85% (non-condensing)

Other requirements:

- Do not place anything on top or lean against the VersaLab.
- Do not place anything on top or lean against the compressor unit.
- Do not operate in wet conditions.
- Do not operate on uneven surfaces.
- Provide natural or mechanical venting and alarm to prevent oxygen deficient atmospheres below 19.5% oxygen.
- Keep self-contained breathing apparatus readily available for emergency use.

C.2.3 Workspace Requirements

For optimum performance the VersaLab will need the following workspace as minimum:

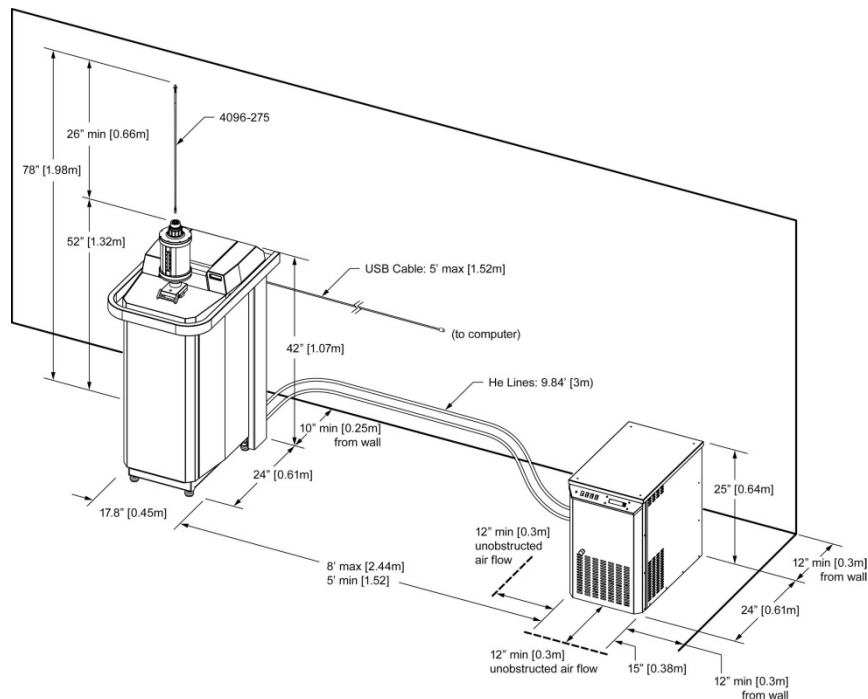


Figure C-1. Workspace Requirements of the VersaLab

C.3 Pin Out Diagrams

This section provides pin out diagrams for VersaLab. Refer to option manuals for pin outs of option hardware, including option pucks and CAN modules.

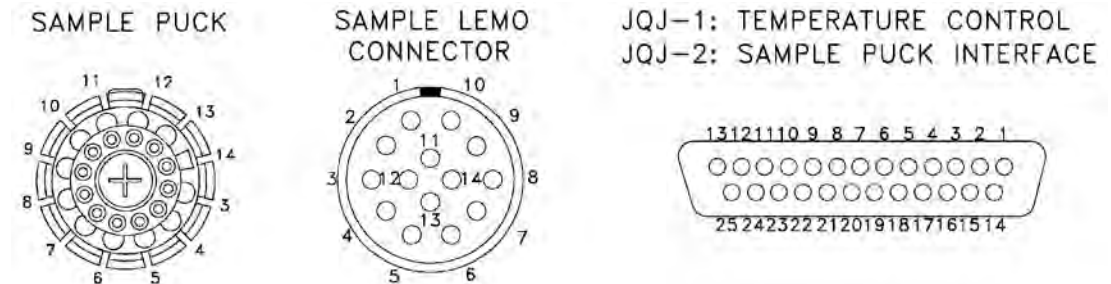


Figure C-2. Sample Puck, sample Lemo Connector, JQJ-1: Temperature Control, JQJ-2: Sample Puck Interface

C.3.1 JQJ-1: Temperature Control

This connector is located on the top of the sample chamber, under the cosmetic stainless steel lid. The spare heater and spare thermometer are not connected.

Table C-1. JQJ-1: Temperature Control Connections

PIN	FUNCTION
2	Spare Heater +
15	Spare Heater -
3	Block Heater +
16	Block Heater -
4	Neck Heater +
17	Neck Heater -
5	Block Thermometer I +
18	Block Thermometer I -
6	Block Thermometer V +
19	Block Thermometer V -
7	Backup Thermometer I +
20	Backup Thermometer I -
8	Backup Thermometer V +
21	Backup Thermometer V -
9	Spare Thermometer I +
22	Spare Thermometer I -
10	Spare Thermometer V +
23	Spare Thermometer V -
11	Serial Com Data MOSI
12	Serial Com Data MISO
13	Serial Com Ground
14	+5 V
24	Serial Com Clock
25	Serial Com Select

C.3.2 JQJ-2 and Sample Puck Connections

The sample connector, which the puck mates with, is located at the bottom of the sample chamber. The sample Lemo connector is located under the plastic module bay lid. The sample D-shell connector is located at the top of the sample chamber under the cosmetic stainless steel lid.

Table C-2. Sample Connections

SAMPLE PUCK	SAMPLE LEMO CONNECTOR	JQJ-2 SAMPLE D-SHELL CONNECTOR	CURRENT CAPACITY (mA)
3	3	5	500
4	4	18	500
5	5	6	10
6	6	19	10
7	7	7	500
8	8	20	500
9	9	8	10
10	10	21	10
11	11	9	10
12	12	22	10
13	13	10	10
14	14	23	10

C.3.3 JQJ-3 Cryostat Connections

The cryostat wiring connector is located on the bottom of the cryostat. It may be easier to access these connections by turning off the power to VersaLab, removing the left side panel, then unplugging the ribbon cable from J18 and probing the ribbon cable. The other end of this ribbon cable is connected to JQJ-3. Be careful not to force typical probes into the ribbon cable connector, as doing so will damage the connector. Use only pins that are designed to fit into the ribbon cable connector. Note that the connector on the end of the ribbon cable is the mirror image of the header.

Table C-3. JQJ-3: Cryostat Connections

PIN	FUNCTION
1	Cryopump Regeneration Heater +
2	Cryopump Regeneration Heater -
3	Second Stage Thermometer V +
4	Second Stage Thermometer V -
5	Second Stage Thermometer I +
6	Second Stage Thermometer I -
7	Magnet Thermometer V +
8	Magnet Thermometer V -
9	Magnet Thermometer I +
10	Magnet Thermometer I -
11	Switch Heater +
12	Switch Heater -
13	Switch Thermometer V +
14	Switch Thermometer V -
15	Switch Thermometer I +

PIN	FUNCTION
16	Switch Thermometer I -
17	Magnet Voltage +
18	Magnet Voltage -
19	First Stage Thermometer V +
20	First Stage Thermometer V -
21	First Stage Thermometer I +
22	First Stage Thermometer I -
23	Spare
24	Spare
25	Spare
26	Spare

C.3.4 JQG-2: User Bridge Connector

This connector provides access to an extra thermometer channel on the controller board.

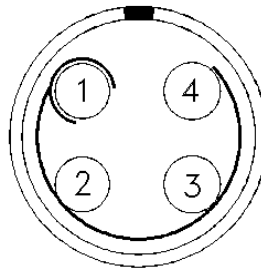


Figure C-3. JQG-2: User Bridge Connector

Table C-4. JQG-2: User Bridge Connections

PIN	FUNCTION
1	I +
2	I -
3	V +
4	V -
Shield	Chassis Ground

C.3.5 JQG-3: QD-CAN Out

This connector is provided on VersaLab to connect future CAN devices, such as an expansion bay. It provides the standard QD-CAN resources, but without ± 24 V power. Devices connected to this port must supply their own power.

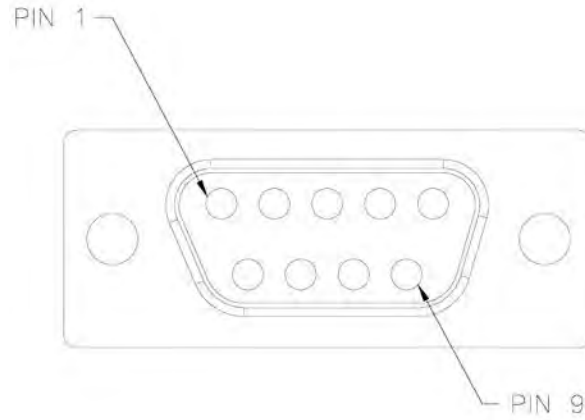


Figure C-4. JQG-3: QD-CAN Out Connector

Table C-5. JQG-3: QD-CAN Out Connections

PIN	FUNCTION
2	CAN Low
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High / Reset

G L O S S A R Y

Glossary of Terms

STANDARD TERM	DEFINITION
Control board	An electronic printer circuit board that controls various VersaLab subsystems. This board resides in the electronics tower.
Electronics tower	Chassis at the rear of VersaLab that houses electronics for control of VersaLab and measurement options. Also houses the diaphragm pump.
Control computer	The computer, running VersaLab MultiVu under Microsoft Windows, which provides an interface to VersaLab for the user.
SROM	Serial Read-Only memory, used to store calibration and configuration information for the piece of hardware it is attached to.
Experimental wiring	Six twisted pairs that run down the outside of the sample chamber, providing connections to the 12 leads on the puck.
CAN module	A module that plugs into the module bay and provides electronic control of a measurement option.
Module bay	Area at the top of the electronics tower that accepts up to four CAN modules. Provides power, CAN, and cooling air to the modules.
Puck	A round disc on which experimental hardware is mounted. Plugs into the bottom of the sample chamber, where it connects to the 12 experimental wires.
System temperature	Temperature of the puck interface as measured by either the block or backup thermometer, depending on how VersaLab is configured.
Option temperature	Sample temperature measured by a thermometer near the sample in a measurement option. Not present on all measurement options.
Control temperature	The temperature that is being used for feedback control. Either the system or option temperature.
Block thermometer	A thermometer mounted on the bottom of the sample chamber whose readings represent the puck interface temperature. See also backup thermometer.
Backup thermometer	Thermometer mounted in same location as block thermometer. Used if block thermometer fails.

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



**Quantum Design Air Cooled Indoor
Helium Compressor User's Manual®**

Part Number 1601-001, A1

Quantum Design

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U.S. Patents

4,791,788 Method for Obtaining Improved Temperature Regulation When Using Liquid Helium Cooling
4,848,093 Apparatus and Method for Regulating Temperature in a Cryogenic Test Chamber
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)
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.
- Section P.5 provides safety guidelines and regulatory information.
- Section P.6 includes RoHS and WEEE statements, as well as disposal information.

P.2 Scope of the Manual

This manual describes the operation of the Quantum Design Helium Compressor. This manual describes the construction and design of the Quantum Design Air Cooled Indoor Helium Compressor, how to setup the Helium Compressor for use, how to use the control panel, and error messages associated with operation and their solutions.

P.3 Contents of the Manual

- Chapter 1 presents a broad overview of the Helium Compressor and its operation.
- Chapter 2 describes the installation of the Helium Compressor.
- Chapter 3 provides information on using the Helium Compressor with the VersaLab system.

P.4 Conventions in the Manual

- File menu** **Bold** text distinguishes the names of menus, options, buttons, and panels appearing on the PC monitor or on the Air Cooled Indoor Helium Compressor (HAC 900) display panel.
- File >>Open** The >> symbol indicates that you select multiple, nested software options.
- `.dat` The `Courier` font distinguishes characters you enter from the PC keyboard. The `Courier` font also distinguishes code and the names of files and directories.
- <Enter> Angle brackets < > distinguish the names of keys located on the PC keyboard.
- <Alt+Enter> A plus sign + connecting the names of two or more keys distinguishes keys you press simultaneously.
- Note** Text is set off in this manner to signal essential information that is directly related to the completion of a task.

CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.



WARNING!

Text is set off in this manner to signal conditions that could result in bodily harm, loss of life, or irreparable damage to equipment.



WARNING!

Text is set off in this manner to signal electrical hazards that could result in bodily harm or loss of life.



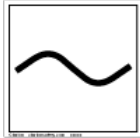
PROTECTIVE CONDUCTOR TERMINAL

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 users 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.



This symbol signals information on **fusing**.


P.5 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 Quantum Design Air Cooled Indoor Helium Compressor (HAC900) 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.


This product is NOT user serviceable except for the operations that are described as performable by the user in Appendix A.

Observe the following safety guidelines when you use your system:


WARNING!	
	<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>

P.5.1 Inspection for Damage

The Air Cooled Indoor Helium Compressor (HAC900) 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.

WARNING!	
	<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.5.2 Pressurized Gas

	WARNING!
	The helium compressors are charged with helium gas at high pressure. Typical pressures found in the system can range from 0.1-2.5 MPa. This requires the operator to follow appropriate safety protocols.

Always wear protective clothing, including eye protection, gloves and covered shoes when you work with pressurized gas. The compressor is equipped with low loss fittings, so attaching and removing helium hoses does not require a total venting of the system.

- Work with high gas pressure lines only in well-ventilated areas. In the event of a gross helium gas leak from the gas lines, vent the room immediately and evacuate all personnel. In a poorly ventilated area, helium can displace the air, leading to asphyxiation. Because helium rises, well-ventilated rooms with high ceilings are generally the safest.
- Arrange the compressor lines, power and connectivity cable, and helium supply lines in a neat manner to avoid tripping hazards.
- Do not pinch the high pressure lines from the helium compressor or bend them with a radius tighter than 60 m (24 inches). These lines are at a pressure of about 15 bar (300 psi). A puncture in the helium lines might cause an explosion and rapid escape of high pressure gas.
- Wear hearing protection whenever performing a cold head vent to avoid hearing loss.

P.5.3 Electricity

	WARNING!
	High voltage is supplied to the compressor making a shock hazard possible if inadequate safety procedures are followed.



- In case of emergency, switch the power off at the back of the helium compressor or unplug the main power cords from the wall power outlets.
- 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. To prevent electrical shock, disconnect the compressor from the power before you install it, adjust it, or service it.
- Keep electrical cords in good working conditions, and replace frayed and damaged cords.
- Do not replace detachable MAINS electrical supply cords by 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.
- In general, keep liquids away from the helium compressor unit.
- Keep the helium compressor away from radiators and heat sources. Provide adequate ventilation to allow for air cooling around the compressor.
- If the compressor is used in a way not specified by Quantum Design, then the protection provided by the equipment may be impaired.
- Be sure to follow circuit breaker specifications outlined in the helium compressor manual.

P.5.3.1 TURNING OFF THE HELIUM COMPRESSOR

- Make sure the helium compressor is tuned off before connecting or disconnecting the cold head cable. Failing to observe this precaution may result in electrical shock.
- Set helium Compressor to “OFF” state by depressing once the “RUN” button on the front control panel. The front panel will then display: “OFF by REMOTE CTRL”. Note that it might take a few minutes for the Cold Head to stop. Once the Cold Head is completely stopped proceed to the next step.
- Power down the indoor compressor by toggling the switch in the back of the indoor compressor control unit. This will shut down any 200-240 V power.
- Disconnect the 200-240 V power by turning off the Low Voltage breaker.
- Unplug the compressor power cords from the power source if needed

P.5.4 Moving the Indoor Helium Compressor

WARNING!	
	Never operate the HAC900 on uneven surfaces or on inclines greater than a 1:12 ratio.
	ALWAYS turn off the helium compressor before to moving it. (See Section P.5.3.1)

The indoor helium compressor has four caster wheels and can be moved relatively easily. The unit should be powered off, prior to moving it, according to the instructions given in the above section. Disconnect any communication or power cables, as well as the compressor return and supply lines. Simply roll the compressor to its new location.



WARNING!

Be careful when moving HAC900 indoor helium compressor. Do not drop the instrument, because doing so could cause damage. Do not attempt to lift the instrument by yourself, because doing so could cause injury. Take care not to damage the high-pressure gas lines when moving the instrument.

P.6 Disposal Information

The Quantum Design Air Cooled Indoor Helium Compressor (HAC900) 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 Quantum Design Air Cooled Indoor Helium Compressor (HAC900) 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.6.1 RoHS Statement

RoHS Statement: The Quantum Design Air Cooled Indoor Helium Compressor (HAC900) is a Research and Development instrument that falls into Category 9 RoHS exemption.

P.6.2 WEEE Statement

WEEE Statement: The Quantum Design Air Cooled Indoor Helium Compressor (HAC900) 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

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

<i>Component Description / Location</i>	<i>Identifying Photograph/Diagram</i>
Display Panel Assembly (QD P/N.: 4601-061-01)	
Compressor Controller Assembly (QD P/N.: 4601-130-01)	
Helium Scroll Capsule (QD P/N: HM504)	
Electronics bay assembly (QD P/N: 4601-160)	

Getting Started

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the chapter.
- Section 1.3 presents the specifications of Helium Compressor.
- Section 1.4 explains the basic functionality.
- Section 1.5 discusses variable speeds of the scroll compressor and cold head.
- Section 1.6 explains the compressor status monitoring.
- Section 1.7 discusses safety.
- Section 1.8 discusses the Quantum Design Air Cooled Helium Compressor Interfaces and Connections.
- Section 1.9 explains operating the Quantum Design Air Cooled Helium Compressor.

1.2 Overview

This chapter will give a brief introduction into the operation of the Quantum Design Air Cooled Helium Compressor (QDHC). It will include a brief description of the compressor, including the layout and design. The chapter will also cover the basic functionality and the operation of the compressor, as well as the monitoring of the compressor using the embedded microcontroller.

1.3 Specifications

The specifications of the Quantum Design Helium Compressor are summarized in Table 1.1.

Table 1-1. Specifications

Dimensions	
Width	14.4"
Length	24"
Height	24.5"
Flex Line Length	10 meters
Helium Gas Pressure	
Static	1.72 to 1.78 MPa
Operating	0.4 (Return Side) to 2.5 MPa (Supply Side)

1.4 Theory of Operation

Cold heads require a supply of clean, compressed helium in order to achieve cryogenic refrigeration. The QDHC compresses helium gas using a scroll compressor capsule to supply helium at pressures up to 2.5 MPa. Since the supplied helium must be free of any oil before entering the attached cold head, as any remaining oil in the pressurized helium may freeze in the cold head and potentially cause serious damage to the cold head, the helium gas passes through two filters and is cleaned before exiting the QDHC. The first of these, the Coalescing Filter, consists of a large number of fiber glass filter elements which filter the helium and coalesce any oil mist that is present. The oil is then collected in an oil sump, which is periodically drained to the compressor capsule. The second filter is a charcoal adsorber. Any oil vapor that passes through the coalescing filter is adsorbed by the charcoal pellets present in the adsorber. All the oil removed by the adsorber remains within it. As a result it must be periodically replaced (after 30,000 hours of operation). After the compressed helium exits the cold head at reduced pressure, it is returned to the low pressure side of the compressor capsule. In order to avoid undesirable low pressure swings on the compressor capsule return, a surge volume is included in the return line. The surge volume also contains a filter to remove any particulate that is carried with the helium before returning to the compressor capsule. See Figure 1 for the location of these components.

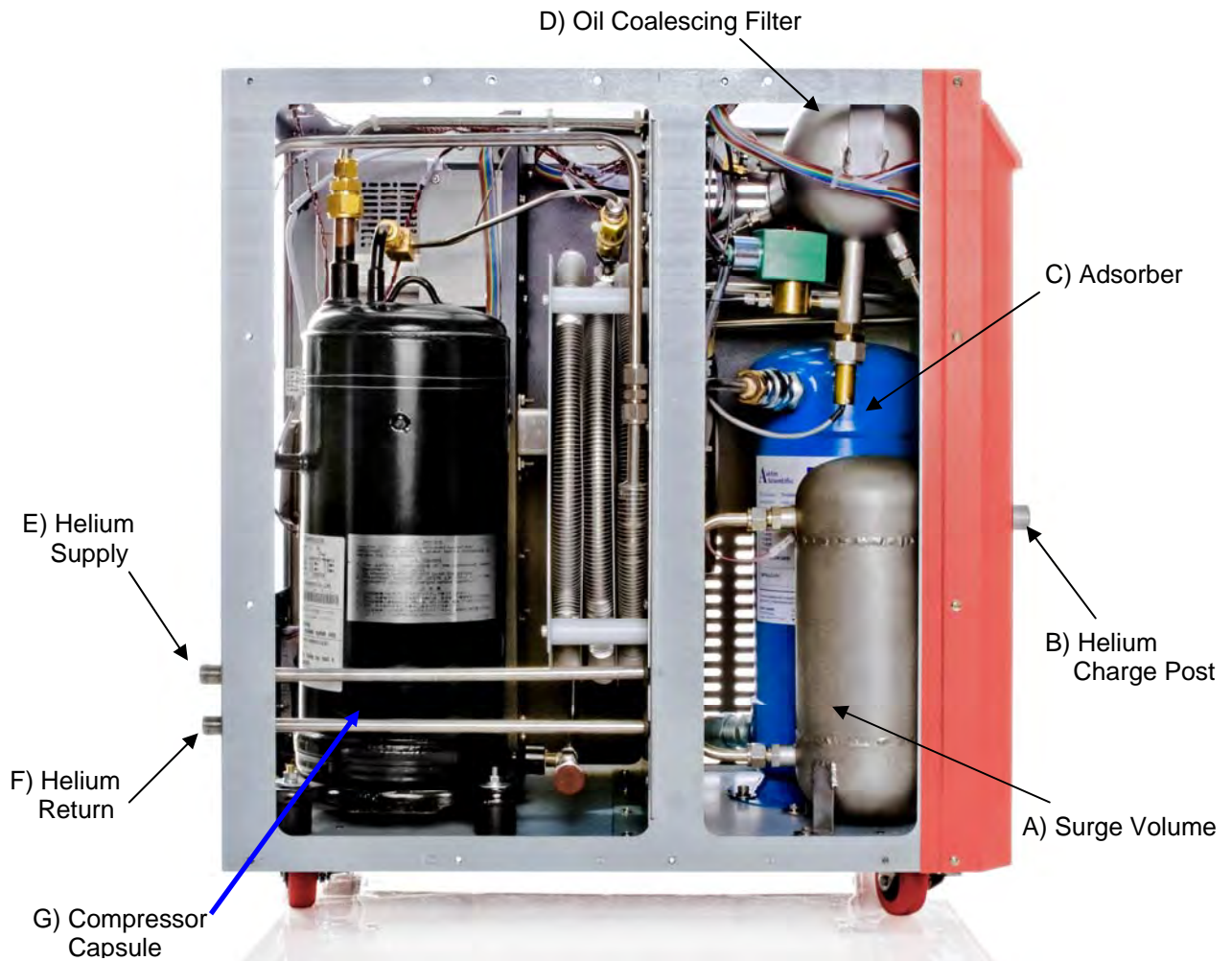


Figure 1-1. Internal structure of the Quantum Design Helium Compressor including: Surge Volume (A), Helium Charge Port (B), Adsorber (C), Oil Coalescing Filter (D), Helium Supply (E), Helium Return (F), Compressor Capsule (G).

1.5 Variable Cooling Power Capability

In order to avoid generating excess cooling power, and therefore wasting electrical power, the Quantum Design Helium Compressor has the capability to vary the cooling power of the cold head. Variable cooling power is accomplished by varying the compressor capsule speed and the cold head motor drive speed. Each of these speeds is controlled using a variable frequency drive (commonly called an inverter). These inverters convert the supplied 220 V single phase power to the required three phase power required by the compressor capsule and cold head motors. The on board microcontroller uses the inverters to control the speeds of the compressor capsule and cold head drive motor to achieve the desired cooling power.

There are three preset power levels available for the compressor – High, Normal, and Low. The speeds at which the compressor capsule and cold head drive motor typically run in these situations are described in Table 1.2. These speeds can be customized, so your compressor might have different preset speeds.

Table 1-2. Compressor Operating Speeds

Power Level	Scroll Compressor Speed	Cold Head Speed
High	30	70
Normal	15	45
Low	12	30

In addition to the three preset power levels the, the Quantum Design Helium Compressor can be fine tuned by the user. By taking manual control of the Helium Compressor speeds, the user can affect the supply and return pressures as well as the cooling power.

1.6 Compressor Status Monitoring Using the Embedded Microcontroller

To ensure the safe operation of the Quantum Design Helium Compressor, a variety of compressor conditions are monitored, including: pressures, temperatures, oil level, and oil flow. The microcontroller can then use these parameters to adjust operating conditions to be as efficient as possible. For instance, the scroll compressor or cooling fan speeds can be modified to prevent over-heating, or the system can open a solenoid valve to allow oil to drain from the coalescing filter sump and return to the scroll compressor at a predetermined value.

Pressure sensors are located at the surge volume (the return pressure) and the coalescing filter (the supply pressure). In addition to ensuring proper supply pressure to the cold head and scroll compressor; the pressure sensors, along with the embedded microcontroller, monitor the difference in pressures as well as the helium charge. This allows the system to determine if a leak exists or the cold head was improperly attached.

The capsule, cool oil, cool helium, and exhaust temperature are also monitored to verify proper cooling and air flow of the entire QDHC. Additionally, the microcontroller estimates oil flow by finding the ratio of the differences in cool oil and cool helium temperatures to the capsule and cool helium temperatures.

The oil level in the coalescing filter sump is monitored using a capacitance sensor, which is continually monitored by the microcontroller. Once a maximum level is reached (or after 20 minutes), a solenoid valve is activated, allowing the oil in coalescing filter to drain to the compressor capsule.

1.7 Safety

Because of the possible dangers associated with the compressor, it is essential that the operator be cognizant of the safety issues associated with the compressor.

WARNING!

Pressurized Gas



The Quantum Design Helium Compressor is compressing Helium gas to high pressures. Typical pressures found in the system can range from several tenths to nearly 3 MPa. This requires the operator to follow appropriate safety protocols. These include venting of the system before it is opened for any reason. (The compressor is equipped with low loss fittings, so attaching and removing flexible hoses does not require a total venting of the system. Provided the pieces to be connected are at similar pressures, simply attach or detach as necessary.)

WARNING!

Electrical



The compressor is supplied with 220 VAC power making a shock hazard possible if inadequate safety protocols are followed. In order to avoid electrocution hazard, do not operate the compressor with any enclosure panels removed.

1.8 Interfacing with the Quantum Design Air Cooled Indoor Helium Compressor

1.8.1 Front Panel

The front panel provides a convenient interface for operation of the QDHC. The switches, together with the display, can provide a wealth of information to the user. Additionally, a low loss fitting, which provides access to the Helium compression system is placed on the front panel.

1.8.1.1 USER INTERFACE

The user interface consists of four switches (run, head, mode, and display) along with an LCD panel. The run switch can be in one of two positions: on or off. This button provides a method for starting or stopping the compressor capsule from the control panel. The head switch will cause the cold head to run by itself for five minutes. This is done in case the cold head needs to be purged of gas. By pressing the mode switch, the operator can cycle through the three preset compressor operating modes – low power, normal power, and high power. The various parameter displays associated with the compressor can be accessed by depressing the display switch (*e.g.*, compressor hours, supply and return pressures, and other useful system parameters).

Note: Refer to Appendix B for a complete diagram of the menu structure.

1.8.1.2 HELIUM FILL

Note: Refer to Appendix A for Maintenance.

In addition to the operation controls found on the front panel, an access port to the helium system can be found on the front body panel. This access port allows the user to pressurize the system or measure the pressure (using an external pressure gauge).

CAUTION!

It is important to note that the front panel helium port should not be used to vent the system, as this will cause helium to flow in an unintended direction through the compressor capsule which could possibly cause damage.

1.8.2 Rear Panel

There are a number of connections on the back panel of the compressor, including power and helium gas connections.

1.8.2.1 LINE VOLTAGE AND BREAKER

Line voltage at a nominal 220 V supplies power to the compressor. Typically this comes from the VersaLab system, but power can be supplied directly to the compressor with an appropriate cable.

1.8.2.2 QD-CAN CONNECTOR

The QD-CAN line provides a communication between the QD compressor, the QD VersaLab system, and the computer control. The various display parameters associated with the compressor are transmitted via this line for analysis and troubleshooting.

1.8.2.3 COLD HEAD DRIVE CABLE

Power to the cold head is supplied through this connection. It is a six pin connector with three connections for three phase AC; one connector for ground and the final two for a thermostat attached to the cold head drive motor.

1.8.2.4 HELIUM SUPPLY AND RETURN LINES

Note: Refer to Chapter 2, Installation, for Connection Instructions.

The two low loss fittings connect the supply (top) connection and return (bottom) connection lines of the compressor to the cold head. These fittings prevent loss of helium charge, but provide a quick connection to the supplied supply and return hoses.

1.9 Operating the Quantum Design Air Cooled Indoor Helium Compressor

1.9.1 Initial Installation

Note: Refer to Chapter 2, Installation.

1.9.1.1 RUNNING AND STOPPING THE COMPRESSOR AND COLD HEAD.

The Quantum Design Helium Compressor is simple to operate. Simply press the run switch and the compressor will start in the current operating mode (or normal mode if the compressor microcontroller has been reset).

Operation of the compressor is simple and has been developed to run with very little oversight when used together with the VersaLab system. Once the compressor has been powered up simply pressing the run switch will start the compressor in the current run mode. Operation of the system can also be controlled remotely via the attached CAN line.

It is very possible the owner may never need to actually operate the compressor directly. Rather all operation of the compressor could come about through the VersaLab system itself.

Stopping the compressor is also simple. The user need only depress the run switch for the compressor and cold head to stop running.

If the user needs to use the compressor for another use other than the VersaLab system, contact QD to discuss with an engineer the most appropriate method for your needs.

1.9.1.2 CONTROLLING THE SPEED OF THE COMPRESSOR AND COLD HEAD

Three separate cooling modes control both the compressor capsule and cold head drive motor speeds. These two speeds are optimized together to provide three levels of cooling power: the high power mode, the normal mode, and the low power mode. The high power provides the greatest cooling power and is generally used for system cooldown and those situations where a greater amount of cooling power is required. The normal mode is for general operation, for it provides enough cooling power for the VersaLab system to maintain design temperatures for the sample, as well as allowing for use of the magnet. The low power mode is generally used for standby conditions when the magnet will not be used, but it is important for the system to remain cold enough to be close to operating conditions.

In addition to the three predefined modes, the compressor can be programmed remotely using the QD-CAN system. This allows the user to independently modify the Cold Head drive motor speed and the compressor capsule speed to find the most optimal arrangement for the current cooling needs.

Once the unit is in remote mode, manual mode can be reestablished by simply pressing the run mode button.

Again, the Quantum Design Helium Compressor has been designed with the VersaLab system in mind. This means that the compressor will be able to supply clean, pressurized helium gas to the VersaLab cold head with minimal oversight of the user, simply plug and go.

1.9.1.3 TYPICAL OPERATING PARAMETERS

Note: The user does not need to monitor these parameters, but are presented here for reference.

For a lab at 25 C, typical operating parameters for the Quantum Design Helium Compressor are listed in Table 1.3. These values are meant only to be a reference; the actual values of your compressor may vary. It is also very typical for the parameters to change through out the day as the ambient temperature varies.

Table 1-3. Typical Operating Parameters

	Typical Values		
	Low	Normal	High
Temperatures (C)			
Capsule	44-54	55-65	60-70
Cool Helium	24-28	26-30	28-35
Cool Oil	40-45	48-52	55-65
Exhaust	35-40	40-44	42-48
Pressures (MPa)			
Supply	1.85	1.9	2.1
Return	0.65	0.6	0.5
Oil Flow Ratio	0.6 – 0.7	0.65 – 0.75	0.7 – 0.8

1.9.1.4 THE OIL FLOW RATIO

The oil flow ratio is defined as $(Cool\ Oil\ T - Cool\ He\ T) / (Capsule\ T - Cool\ He\ T)$.

The oil flow ratio (OFR) gives a good indication of how well the oil is flowing as well as if it is being cooled in the system. For instance if OFR = 1, then the oil is not being cooled properly in the oil cooling system, since Cool Oil T is equal to the Capsule T. At the other extreme, OFR = 0, then the Cool Oil T is equal to the Cool He T, which is an indication of no oil flow, or an inadequate oil charge.

Installation

2.1 Introduction

This chapter contains the following information:

- Section 2.2 presents the chapter overview.
- Section 2.3 explains the safety requirements.
- Section 2.4 explains attaching the supply and return lines to the cold head.
- Section 2.5 explains attaching the 220V power cable.
- Section 2.6 explains connecting the cold head drive cable to the compressor.
- Section 2.7 explains the attachment of CAN to the QDHC.

2.2 Overview

This chapter describes how to install the Quantum Design Helium Compressor. The first section describes safety. Read this section first to avoid injury. This chapter also describes how to connect the compressor to a cold head, and how to connect QD-CAN to the compressor for communication with VersaLab.

2.3 Safety

To ensure personal safety and proper operation of the Helium Compressor, the power requirements of Appendix C must be followed. A power supply that does not conform to these requirements could result in damage to the system or electric shock to the operator. Be sure the main power breaker is open before continuing with the installation.

The Helium Compressor comes pre-charged with about 250 psig of Ultra High Purity (UHP) Helium gas. It is therefore important to take proper precautions when connecting or disconnecting hoses.

Note: Refer to Appendix C for system requirements.

All the connections are on the back panel of the compressor. Tools that will be needed in the process include: the supplied wrenches, a Phillips head screwdriver, and soapy water.

2.4 Connect the Helium Lines

The helium lines are easily installed to the compressor and VersaLab system. Before installing it is important to check the cleanliness of the connections to ensure that particulates do not enter the closed helium system as this can lead to damage to the cold head, the compressor system, or both.

Using the supplied wrenches attach the return line followed by the supply line. It is also advised that the back panel be removed and a backing wrench be used on the braze fitting to prevent this from becoming too loose and cause the system to vent. Once the lines are attached, a simple leak check using soapy water on the fittings is advised.

2.5 Connect the Power

Make sure the power input module is in the open position – the circle depressed. Connect line power from a wall outlet or the VersaLab system to the power input on the compressor.

2.6 Connect the Cold Head Drive Cable

Attach the cold head drive cable from the compressor to the cold head. This is a six pin connector, so the orientation is important. Make sure the key of the connector fits the slot of the cable.

2.7 Connect the CAN Line (if used)

If you plan to control the compressor via QD-CAN communication, attach the CAN cable to the JQK-1 QD-CAN connector on the back panel. The VersaLab system requires QD-CAN to be connected to the compressor, so be sure to make this connection on VersaLab systems.

Using the Quantum Design Air Cooled Indoor Helium Compressor with VersaLab

3.1 Introduction

This chapter contains the following information:

- Section 3.2 presents an overview of the chapter.
- Section 3.3 discusses compressor operation when used with VersaLab.

3.2 Overview

Chapter 3 will give details on the operation of the Quantum Design Helium Compressor used with the VersaLab system.

3.3 Compressor Operation (other than run/stop) handled by VersaLab: Little Interaction for User

When the Quantum Design Helium Compressor is used together with the VersaLab System, operation generally amounts to pressing the Run switch. The VersaLab controller and software take care of detailed operation of the compressor once you have set it to run.

CAUTION!

The Helium compressor supplies the VersaLab with pressurized Helium gas as well as the power for the Cold Head drive motor, so please ensure these connections are correct.

Check that the CAN connecting the VersaLab and the Quantum Design Helium Compressor is in place, as this is how communications between the two takes place.

Once the system is running, VersaLab controls the compressor and cold head speeds based on the current cooling requirements. This includes Standby vs Active Modes, as well as the sample chamber requirements. Important Diagnostic information is also recorded in the background of the VersaLab Multivu allowing a QD service individual to quickly analyze any problems.

Note: Refer to VersaLab Manual for instructions to operate VersaLab.

Maintenance and Servicing

A.1 Introduction

This chapter contains the following information:

- Section A.2 presents an overview of the Maintenance and Servicing Appendix.
- Section A.3 discusses charging the system with Helium gas.
- Section A.4 explains the lifetime of the adsorber and its service.

A.2 Overview

A general discussion of the required maintenance will be given in this chapter. Although, the Quantum Design Helium Compressor is largely free of user interaction once it is running, there are a number of things the user can do to ensure long term operation.

A.3 Helium Fill

The Quantum Design Helium Compressor comes precharged with about 1.75 MPa of Ultra High Purity Helium, yet it may be necessary for the user to recharge the unit in the case of an accidental vent. When pressurizing the system, 1.45 MPa must be charged to the unit for it to operate, but the ideal pressure to fill the system is $\sim 1.75 \pm 0.03$ MPa (254 \pm 4 PSI). The necessary equipment for this are: 1) a regulator capable of outlet pressures greater than 300 PSIG, 2) a fill line with a female RF fitting – we use RF 04 FN fittings from Faster Inc. 3) a purge assembly to reduce the pressure in case the charge is too high, and 4) Ultra High Purity Helium.

CAUTION!

It is important to use Ultra High Purity Helium, as the moisture content is lower (< 2ppm) reducing the chance of water vapor freezing in the cold head, and causing potential damage to the system.

If it is necessary to purge the system, be sure to attach the purge line to the supply line of the helium compressor.

CAUTION!

Attaching the purge line to the front fill port or the return line could cause helium to flow in an unintended direction, which could cause damage to the compressor.

When filling the compressor, there are two options available. The first and simplest is to use the fill line located on the front panel of the Helium compressor. The second option is to attach to the return line connection on the rear of the compressor. The return line is used for this, again, to ensure proper flow direction of Helium through the compressor.

A.4 Adsorber Change

The Adsorber is a crucial component of the Quantum Design Helium Compressor, as it removes any oil vapor present in the compressed Helium before passing to the cold head. The adsorber lifetime under typical usage is rated at 30,000 hours, so the microcontroller will not allow the compressor to run past this time without an adsorber change.

Contact your Quantum Design service representative to discuss replacement of the adsorber.

Firmware Reference

B.1 Introduction

This chapter contains the following information:

- Section B.2 presents an overview the firmware the monitors and controls the QDHC.
- Section B.3 explains the user interface references.
- Section B.4 explains error messages.

B.2 Overview

This section gives a basic overview of the Quantum Design Helium Compressor firmware, details of the front panel switches, and various causes of error conditions with required action by the user.

B.3 User Interface Reference

There are four switches on the front panel.

1. RUN - The run switch is modal and is used to set the compressor to running or not running. The RUN switch is in series with a virtual RUN switch associated with CAN. Both must be enabled for the compressor to run. This allows the user to have the compressor in an on state, but the firmware the capability of starting or stopping the compressor as needed.
2. HEAD - This switch is momentary and used to run only the cold head for 5 minutes. It is only active when the compressor capsule is not running. Fore instance, if the user needs to run only the head, for cooler maintenance, this switch is pressed.
 - a. The Head Motor runs at 30 Hz (or the current programmed value).

- b. The Head Motor runs for 5 minutes, then turns off.
- c. The Head Motor can be turned off at any time during this operation, by pressing the HEAD switch a second time.
- d. While the head is running the display will read:

Line 1: Cold Head Running.

Line 2: for MM:SS (a countdown time).

- 3. MODE – This switch is momentary and is used to cycle between the various operating modes of the compressor. The power modes cycle as:

Normal Power -> High Power -> Low Power -> Normal Power ...

- 4. DISPLAY – This switch is momentary and is used to cycle through the various display modes. When there is not an alarm, the display will be in one of its standard modes. By pressing the DISPLAY switch, the display will be toggled between the modes described below. (See Figure B-1.) The display will return to the default mode after 10 seconds. There are two lines to the display.

A. Default Mode

a. Line 1

- i. The Left side will display whether the compressor is Running or Off.
- ii. The right side will display how the compressor is being controlled: Remote or Panel Ctrl.
- iii. If the RUN switch is on, but the RUN state is disabled remotely, “Disabled by Remote” will be displayed.

- b. Line 2 will display the Power mode (Low Power, Normal Power, or High Power) or the set speeds (Comp ## Hz Head ## Hz) if the compressor is in remote mode.

B. Adsorber Hours mode

- a. Line 1 displays the number of hours the Adsorber has been used – Adsorber AA,AAA Hrs.
- b. Line 2 displays the number of hours remaining before the Adsorber needs to be replaced – Remaining RR,RRR Hrs. Currently, this is based on a 30,000 hour adsorber lifetime.

C. Compressor Hours Mode

- a. Line 1 will display the number of hours the compressor has been running – Compressor CC,CCC Hrs.

- D. Diagnostic Mode: This mode provides various diagnostic parameters for the user.

- a. Line 1 gives Return and Supply pressures, oil level, and oil flow ratio as; R.RR/S.SS O 00% F.FF
 - i. R.RR = Return pressure (MPa)
 - ii. S.SS = Supply Pressure (MPa)

- iii. O 00% = Oil Level (%)
 - iv. F.FF = Oil Flow Ratio
- b. Line 2 gives the various temperatures at the system: C cc H hh O oo E ee
- i. cc = Capsule Temp (C)
 - ii. hh = Cool Helium Temp (C)
 - iii. oo = Cool Oil Temp (C)
 - iv. ee = Exhaust Air Temp (C)

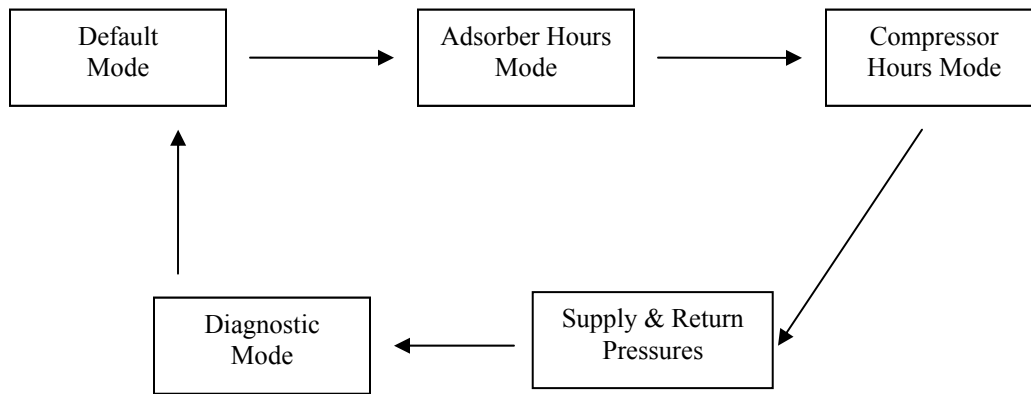


Figure B-1. The display modes of the Quantum Design Helium Compressor. The arrows indicate the next display type that can be accessed by pressing the display switch. Fill Mode can only be accessed while the compressor is not operating.

B.3.1 Audible Alarm

There is an audible alarm that alerts the user to faults, malfunctions, or service needed in the near future.

B.4 Error Message Reference

Below is a list of the error messages associated with the Quantum Design Helium Compressor, along with possible causes and solutions or maintenance that could be used to correct the problem.

The error dictionary below includes the **error message** given on the display, followed by the cause. After which is given likely causes and possible solutions.

1. **Return Pres Too Low** - This fault occurs when the return pressure is below its minimum set point 0.35 MPa. The most likely causes for this would be the Cold Head stopped running or the return line is detached. Appropriate actions would be to check the return line and cold head.
2. **Supply Pres Too High** - This fault occurs when the supply pressure exceeds 2.75 MPa. It is caused by similar conditions as the Return Pres Too Low fault – the Cold Head stopped running or, in this case, the supply line is detached. In general, the Return Pres Too Low fault will occur before this fault, but actions include checking the cold head and supply lines.
3. **Delta Press Too Low** - If the differences between the supply and return line pressures falls below 0.3 MPa will cause this fault. This would be indicative of a plumbing problem. A problem occurred in the cold head causing the supply and return lines to approach a common value. Check that the supply and return lines are attached correctly and not switched.
4. **Capsule Over Temp** - This fault occurs when the capsule temperature exceeds 85 C. A fault of this type could indicate improper oil flow or helium flow. It also could be caused by vents on the compressor being blocked. Check the Oil Flow Rate to get a sense of the oil flow through the system. Check that air flow through the compressor is not being inhibited by any external object.
5. **Helium Over Temp** - If the Cool Helium temperature exceeds 50 C a Helium over temperature fault will be displayed. Like the capsule over temp fault, this is probably due to insufficient air flow through the cooling system. Check that the vents are clear of debris and that the fan is running.
6. **Exhaust Over Temp** - When the exhaust temperature of the cooling system exceeds 55 C, the exhaust over temperature fault will be displayed. Again, this is most likely due to a problem with flow of air through the cooling system. Check for any thing impeding air flow and that the fan is running properly.
7. **Low Oil Flow** - If the oil flow ratio falls below 0.4, a low oil flow fault will occur. This alarm is telling you that the flow of oil through the oil cooling coil is insufficient. Since the oil flow depends on the differential pressure of the system, this alarm is commonly caused by a low differential pressure. Check that the supply and return lines are properly attached, and there is nothing that is causing a bypass of the cold head. Additionally, the differential pressure across the cold head depends on both the speed of the cold head motor and the compressor speed. A very high cold head speed could cause a drop in the differential pressure as could a very slow compressor speed. However, this scenario is highly unlikely.
8. **Oil Sensor Failure** - If there is a problem in the oil sensor electronics this fault will occur. This is usually due to the oil sensor line being unplugged. Check that the oil sensor is attached to the board. Additionally, this alarm could be caused by a short somewhere in the sensor. If this is the case, contact Quantum Design Service.
9. **Oil Return Failure** - When the Oil level fails to fall below 30 % in 30 seconds, an oil return failure will be produced. This is indicative of a problem in the oil return line, or in the oil level sensor itself. Give the system a chance to settle and try again. If the problem continues, contact Quantum Design Service.
10. **Adsorber Full** - After 30,000 hours of operation, the adsorber full fault will inform you that the adsorber needs to be replaced. Prior to this fault occurring there will be

several warnings given to the user indicating a change may be necessary soon. Contact Quantum Design Service to schedule this replacement.

11. **High Oil Level** - If the oil return system is failing and the oil level continues to increase this fault will occur when the Oil level fills the sump. Cycle the compressor power and restart the system. Contact Quantum Design Service if this alarm continues.

System Requirements, Internal Diagram, and Pin-out Diagrams

C.1 Introduction

This chapter contains the following information:

- Section C.2 presents an overview of Appendix C.
- Section C.3 explains the system requirements.

C.2 Overview

The purpose of this appendix is to give the user an overview of the system requirements, pin-outs, and diagrams of the internal systems.

C.3 System Requirements

The system requires 220 V single phase AC of 50 or 60 Hz. If the system is used in conjunction with a VersaLab system, the power for the compressor will be supplied from the VersaLab system. For other cases the user must supply the necessary power requirements for the compressor operation.

The compressor is designed for indoor use only. Normal operation is for typical laboratory conditions, 35 C. Although, the compressor is able to operate in higher ambient temperatures, the capabilities will be reduced, as the increased ambient temperature results in reduced cooling capacities of the air cooled system.

It is also important to keep in mind that the compressor needs adequate air flow. Give the compressor sufficient room for air flow (at least 6 inches on each side), and do not have exhaust air from other systems to close.

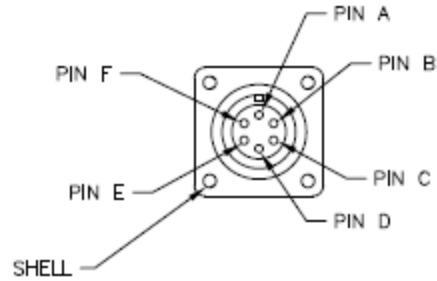


Figure C-1. Cold Head Drive Pin-Out Male

Pin A – Terminal 2 (V) of inverter

Pin B – Terminal 3 (W) of inverter

Pin C – Terminal 1 (U) of inverter

Pin D – Ground and Cable Shield

Pins E and F – Cold Head Drive Motor Thermostat

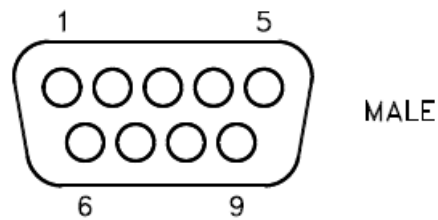


Figure C-2. CAN Connector Pin-Out

Table C-1. QD CAN connector on the rear of the Quantum Design Helium Compressor.

PIN	FUNCTION
1	-24 V DC
2	CAN Low
3	Power Return (24 V)
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High/Reset
9	+24 V DC

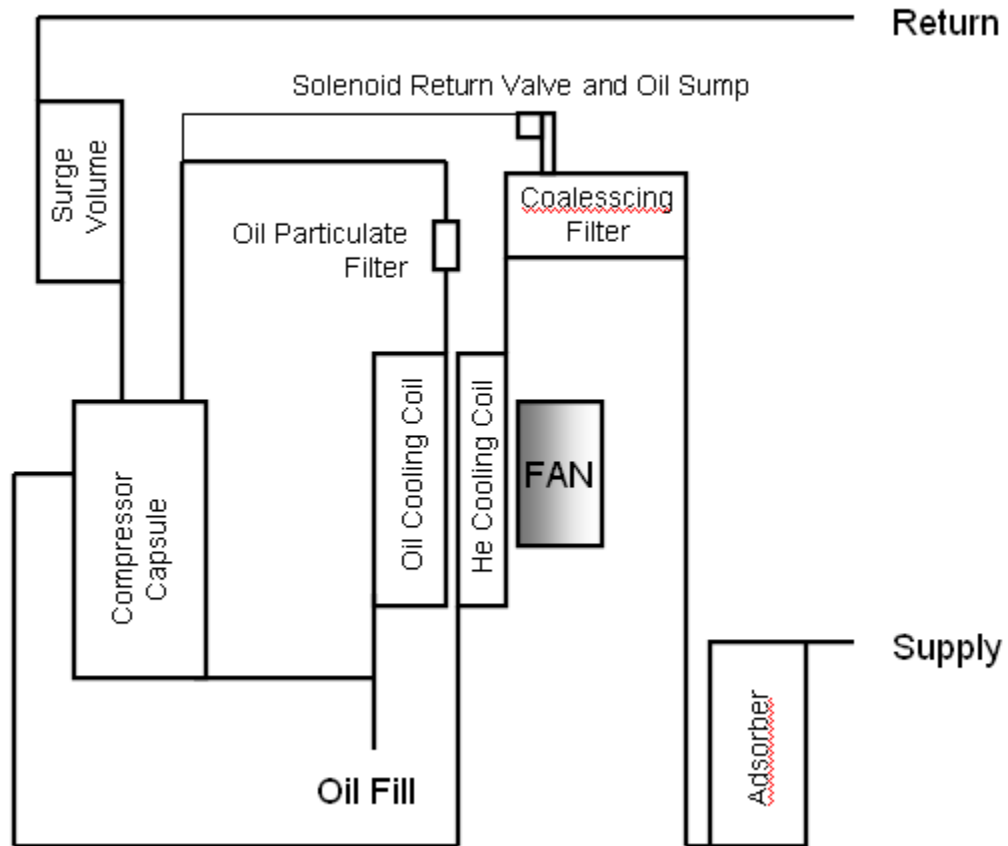


Figure C-3. Plumbing Diagram: Block Diagram 1.

Table C-2. Compressor Diagram Description

Component	Use
Compressor Capsule	Compresses Helium, the Helium output contains oil mist and vapor as well as Helium
Oil Fill	An RF connection used to supply oil for the compressor capsule. The total fill is ~ 1.6 l.
Oil Cooling Coil	Provides a large surface area to cool the oil for adequate lubrication and cooling of the compressor capsule
Helium Cooling Coil	Cooling coil to cool the Helium gas to near ambient temperature. This reduces the vapor pressure of the oil as well making the coalescer more efficient.
Oil Particulate Filter	Removes particulates down to 100 microns.
Fan	Provides air flow over the Helium and Oil cooling coils.
Coalescer	Filters Helium gas and Coalesces Oil mist.
Sump and Solenoid Drain	Oil from coalescer is collected here. When level is sufficiently high, the oil solenoid will open allowing the oil to drain to the compressor capsule.
Adsorber	Removes any remaining oil vapor from helium supply.
Supply/Return	Provides connections to cold head.
Surge Volume	A large volume that provides adequate surge space for venting phase of cold head.

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



Physical Property Measurement System[®]

Heat Capacity Option User's Manual

Part Number 1085-150, Rev. M6

Quantum Design

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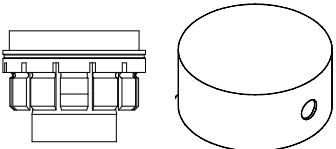
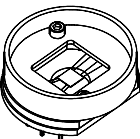
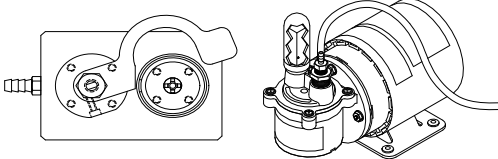
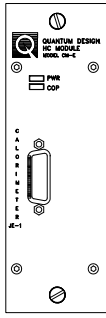
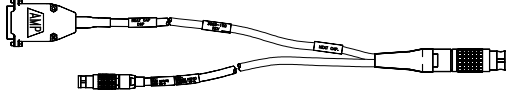
U.S. Patents

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)
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

Components of the Heat Capacity Option

Item	Description	Qty
 <p>Calorimeter Puck and Radiation Shield</p>	<p>Contains sample-mounting platform. Heater and thermometers on puck control and monitor temperature of sample. Puck wires provide thermal link to sample platform.</p>	2
 <p>Spare Puck Frame</p>	<p>Part of puck. Contains puck wires and sample platform. May be attached to puck if wires on original puck frame break.</p>	1
 <p>Sample-Mounting Station</p>	<p>Uses puck interlock and vacuum suction to stabilize puck during sample mounting and removal.</p>	1
 <p>Heat Capacity CAN Module</p>	<p>Contains electronics that read puck platform thermometer and drive puck platform heater.</p>	1
 <p>Cable Assembly</p>	<p>Connects Heat Capacity Controller card to probe.</p>	1

C O N T E N T S

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.

P.2 Scope of the Manual

This manual describes the operation of the Physical Property Measurement System (PPMS) Heat Capacity option for all Quantum Design PPMS-like systems, including PPMS, VersaLab, and DynaCool. It also covers the use of the option with the Helium-3 and Dilution Refrigerator inserts. This manual describes how to install the Heat Capacity option, the purpose and function of the Heat Capacity option, and how to use the hardware and software that are unique to the Heat Capacity option. This manual assumes you are familiar with the Windows operating system.

For detailed information about the MultiVu application, which is the software running the system, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual* or the relevant manual for your system.

P.3 Contents of the Manual

- Chapter 1 presents an overview of the Heat Capacity option.
- Chapter 2 contains installation procedures and explains how to take the first addenda measurement.
- Chapter 4 describes the Heat Capacity software, Heat Capacity analysis techniques, and data file format.
- Chapter 5 explains how to calibrate a calorimeter puck and discusses calibration files.

- Chapter 3 describes the hardware used with the Heat Capacity option.
- Chapter 7 explains how to measure the heat capacity of a sample.
- Chapter 8 explains how to use the Heat Capacity option with the Helium-3 system.
- Chapter 9 explains how to use the Heat Capacity option with the Dilution Refrigerator system.
- Chapter 6 explains how to measure the addenda and discusses addenda tables.
- Chapter 10 explains how to use the Heat Capacity option with the VersaLab System.
- Chapter 11 contains troubleshooting and maintenance procedures for hardware.
- Chapter 12 contains troubleshooting suggestions for measurements.

P.4 Conventions in the Manual

- File menu** **Bold** text distinguishes the names of menus, options, buttons, and panels appearing in MultiVu or the option software.
- File >>Open** The >> symbol indicates that you select multiple, nested software options.
- `.dat` The `Courier` font distinguishes characters you enter from the PC keyboard. The `Courier` font also distinguishes code and the names of files and directories.
- <Enter> Angle brackets <> distinguish the names of keys located on the PC keyboard.
- <Alt+Enter> A plus sign + connecting the names of two or more keys distinguishes keys you press simultaneously.
- Note** Text is set off in this manner to signal essential information that is directly related to the completion of a task.

CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.



WARNING!

Text is set off in this manner to signal conditions that could result in bodily harm, loss of life, or irreparable damage to equipment.



WARNING!

Text is set off in this manner to signal electrical hazards that could result in bodily harm or loss of life.

Overview of the Heat Capacity System

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the Heat Capacity option.
- Section 1.3 discusses the purpose of measuring heat capacity.
- Section 1.4 explains the scope of the Heat Capacity option.
- Section 1.5 discusses special features of the Heat Capacity option.

1.2 What the System Measures

The Quantum Design Heat Capacity option measures the heat capacity at constant pressure

$$C_p = \left(\frac{dQ}{dT} \right)_p.$$

As with other techniques for measuring heat capacity, the Quantum Design Heat Capacity option controls the heat added to and removed from a sample while monitoring the resulting change in temperature. During a measurement, a known amount of heat is applied at constant power for a fixed time, and then this heating period is followed by a cooling period of the same duration.

A platform heater and platform thermometer are attached to the bottom side of the sample platform. (See Figure 1-1 on the following page). Small wires provide the electrical connection to the platform heater and platform thermometer and also provide the thermal connection and structural support for the platform. The sample is mounted to the platform by using a thin layer of grease, which provides the required thermal contact to the platform.

The integrated vacuum system in the cryostat provides a sufficient vacuum so that the thermal conductance between the sample platform and the thermal bath (puck) is totally dominated by the conductance of the wires. This gives a reproducible heat link to the bath with a corresponding time constant large enough to allow both the platform and sample to achieve sufficient thermal equilibrium during the measurement.

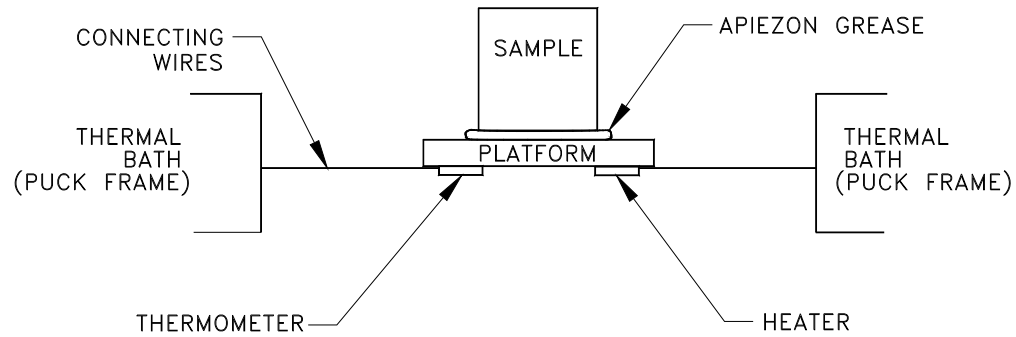


Figure 1-1. Thermal Connections to Sample and Sample Platform in PPMS Heat Capacity Option

1.3 Purpose of Measuring Heat Capacity

The measurement of the heat capacity of solids can provide considerable information about the lattice, electronic, and even magnetic properties of materials. Heat capacity measurements, particularly when taken at temperatures that are well below the Debye temperature, directly probe the electronic and magnetic energy levels of a material and hence allow comparisons between theory and experiment. While electronic transport measurements, such as resistivity, are substantially more common, the link between experiment and theory is not always as clear as it is in a heat capacity measurement. Any statistical theory of matter involves computing the density of states and energy levels; these computations naturally lead to predictions of heat capacity numbers.

From a practical point of view, materials used in the construction of thermal devices, such as refrigerators, cryostats, and so on, must be characterized thermally. Knowledge of the heat capacity of construction materials is important to any successful thermal design.

1.4 Scope of the Heat Capacity Option

1.4.1 Sample Size and Thermal Characteristics

In the Heat Capacity option, the basic puck configuration accommodates small, but not microscopic, samples weighing approximately 1 to 200 mg. Given the thermal characteristics of the calorimeter, this range of masses produces, for most solids, varying relaxation time constants that may be a fraction of a second at 1.9 K or many minutes at 300 K. A single heat capacity measurement can require nearly 10 time constants for the settling time that occurs between measurements. Measuring very large samples can thus be prohibitively time consuming. The addenda heat capacity, however, limits the size of the smallest samples. Measurement accuracy, which is generally a percentage of the total heat capacity, is sacrificed when the sample heat capacity is small compared to the addenda heat capacity.

Since the technique used for measuring heat capacity, as described below in Section 1.4.3, is dynamic in nature, the geometry and thermal diffusivity of the sample must be such that the thermal diffusion time in the sample is small compared to the time constant of the measurement. In cases where the amount of time it takes for the sample to reach internal thermal equilibrium is not small compared to the measurement time, the resulting heat capacity measurement will be too small. Although this problem is indicated in the software as a poor thermal contact between the sample and the sample holder, it is important to use samples that have relatively fast thermalization times to get the correct heat capacity numbers. In cases where the thermal diffusion time in the sample is large, it is necessary to use samples that have a relatively flat geometry, so as to reduce the thermal path through the sample.

1.4.2 Temperature Range

The Heat Capacity option has no fundamental temperature range limit other than the base system (PPMS, DynaCool, or VersaLab) temperature range. However, relaxation techniques are traditionally used at temperatures that are below approximately 100 K, because relaxation times are relatively short below 100 K. The Heat Capacity option can measure heat capacity up to about 400 K.

The percent resolution of the thermometry is relatively constant over the temperature range. Hence, at higher temperatures, the absolute temperature resolution is somewhat poorer.

1.4.3 Measurement Technique

Many different measurement techniques (Stewart 1983) are optimized for different sample sizes and accuracy requirements (high resolution versus accuracy). The Quantum Design Heat Capacity option uses a relaxation technique that combines the best measurement accuracy with robust analysis techniques. After each measurement cycle—which is a heating period followed by a cooling period—the Heat Capacity option fits the entire temperature response of the sample platform to a model that accounts for both the thermal relaxation of the sample platform to the bath temperature and the relaxation between the sample platform and the sample itself (Hwang, Lin, and Tien 1997). The effect of the relaxation between the platform and sample must be considered when the thermal connection shared by the sample and platform is poor. By modeling this effect, the software can report the correct heat capacity values despite such poor contact.

The fitting technique, which is the primary means of data analysis provided with the Quantum Design Heat Capacity software, assumes that the heat capacity is approximately constant over the range of temperatures covered by a single measurement cycle. When the heat capacity has strong variations with temperature, as in the case of first-order transitions, one can use advanced slope analysis techniques provided by the software to resolve rapid variations of heat capacity with a single relaxation cycle.

1.4.4 Pressure in Sample Chamber

The wires connecting the sample platform to the puck frame create well-controlled thermal links to the thermal bath. To eliminate alternate thermal links through residual gas, the pressure within the probe must be less than approximately 1 mTorr. The high-vacuum system, which operates in conjunction with the Heat Capacity option, maintains this low pressure. A charcoal holder is used as a cryopump to help decrease the pressure at the bottom of the probe at temperatures below 10 K. When the High-Vacuum system is activated, base pressures of approximately 0.01 mTorr are typical at the top of the probe.

1.5 Special Features of the Heat Capacity Option

1.5.1 Acquisition Hardware

Relaxation techniques require accurate time-resolution of the temperature response of the sample platform during the measurement cycle as well as precise correlation of the heater output and the temperature response. Fast, accurate thermometry is essential for the best signal-to-noise ratio. These requirements for relaxation calorimetry place considerable demands on the data acquisition portion of a system. Rather than attempt to adapt existing data acquisition hardware to the task, Quantum Design has developed a high-performance controller optimized for relaxation calorimetry.

1.5.2 Calorimeter Puck and Sample Mounting

As with other measurement options, the calorimeter is a puck that you insert into the sample chamber. The Heat Capacity option includes more than one puck, so you can prepare a second sample on the second puck while the first puck is in the sample chamber. When you have measured the first sample, you may immediately insert the second puck and measure the second sample without having to remove the sample from the first puck.

You use a sample-mounting station to hold the puck when you mount and remove samples. The station uses a puck interlock arm and vacuum suction to stabilize the puck and the sample platform. By stabilizing the sample platform, the station helps protect the delicate, thermally conducting wires that connect the platform to the puck frame.

1.5.3 Puck Calibration

The Heat Capacity software includes a fully automatic thermometer calibration routine that uses the base-system thermometer as the reference thermometer to produce temperature calibrations for the puck thermometer, platform thermometer, and platform heater. The calibration routine thus reduces the cost of the pucks, because the pucks do not require factory calibration. Moreover, the calibration routine allows you to design custom pucks that have different characteristics but that can still work with the Heat Capacity option if you use the standard calibration procedure to calibrate the pucks.

1.5.4 Automation Environment

You may program the Heat Capacity option, just as you may program other measurement options, to automatically acquire data. You use sequences, which are the system automation language, to run any number of measurement macros. You may also run each macro independently.

Sophisticated data analysis, which is part of the Heat Capacity option, is critical to your ability to run heat capacity measurements while you are away from the system. Monitoring each measurement and its associated fit in order to detect potential problems is unnecessary. The Heat Capacity software writes all relevant diagnostic information and the heat capacity numbers to an open data file. When the measurement is complete, you examine this data file. Errors are automatically computed for each sample heat capacity measurement.

Installation and Getting Started

2.1 Introduction

This chapter contains the following information:

- Section 2.2 lists system requirements for the Heat Capacity option.
- Section 2.3 explains how to install the Heat Capacity hardware and software.
- Section 2.4 explains how to perform the first addenda measurement.

2.2 System Requirements

- Computer running a current version of the MultiVu software. Current versions can be found at www.qdusa.com.
- Cryopump or Turbo Pump High-Vacuum system.

2.3 Installing the Hardware and Software on a PPMS

This section describes the first-time installation of a new Heat Capacity Option applicable for Standard PPMS, Helium-3 insert, and DR insert. Please see Chapters 8 or 9 in this manual for any additional installation requirements for the Helium-3 or DR inserts. For VersaLab, follow section 10.3. For DynaCool, see Chapter 11.

Before the Heat Capacity option can be installed, either the Turbo Pump or the Cryopump High-Vacuum option must be installed. Refer to your High-Vacuum option manual for detailed instructions.

In the following subsections, you will install or configure the following components:

- Heat Capacity Control Electronics
- Heat Capacity Cable
- Sample Mounting Station
- Heat Capacity Option Software for MultiVu

Detailed descriptions of the hardware are contained in Chapter 3. Insert-specific hardware is described in chapters 8 and 9. The software is described in Chapter 4.

2.3.1 Install the Software

The software may already be installed on your PC. If it is not, or if it is an older version, you should obtain installation CDs or download the appropriate installers from the Quantum Design website (www.qdusa.com).

1. Install MultiVu version 1.4.0.9 or later on the target PC by running Setup.exe from the provided installation CDs or download package.
2. Install Heat Capacity version 3.5.0 or later on the target PC. Be sure to select the **Heat Capacity for He3** or **Heat Capacity for DR** checkboxes during the installation wizard if you have these inserts.

If there are any previous versions of either MultiVu or Heat Capacity, these will be upgraded by the installer.

Note: Version 3.X of the Heat Capacity software is not backward compatible with versions 2.X or earlier. Once you upgrade, you will not be able to go back. Also, Heat Capacity version 3.X will not function with version of MultiVu older than 1.4.0.9.

2.3.2 Install the Heat Capacity Control Electronics and Cable

2.3.2.1 INSTALL THE MODULE 1000 TOWER

If the Model 1000 Tower and the associated CAN network connection has not yet been installed between the PC and the Model 1000, then do that now. Refer to the *Model 1000 Modular Control System User's Manual* for specific instructions.

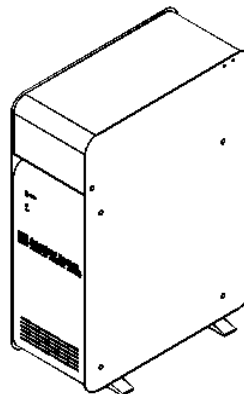


Figure 2-1 External View of the Model 1000 Modular Control System (4100-001)

2.3.2.2 INSTALL THE CM-E HEAT CAPACITY CONTROLLER MODULE

The control electronics for the Heat Capacity option are contained in the Model CM-E Heat Capacity Module. This controller module normally resides in the Model 1000 Tower and may already have been installed.

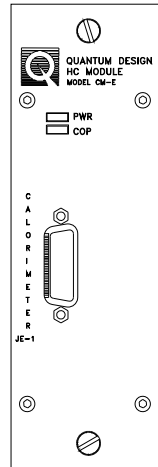



Figure 2-2 Heat Capacity CAN Module

If the Heat Capacity Module is not already installed in the Model 1000, then do this now. The Heat Capacity module does not have any specific cooling or power requirements and can operate in any available module bay in the Model 1000.

1. Turn off the power to the Model 1000.
2. Remove the cover plate from one of the unused module bays and insert the Model CM-E Heat Capacity Module into the empty bay.



WARNING!

The power must be turned off. Hot-plugging a module can destroy it.

3. Once the module is inserted and the mounting thumbscrews screwed in, turn on the power to the Tower. Once the self-test is complete, the PWR LED will be lit solid green and the COP LED will be solid or blinking green. This indicates a normal startup.

2.3.2.3 CONNECT THE CONTROLLER CABLE

Standard Heat Capacity and Heat Capacity with the Helium-3 Insert both use the same Controller Cable (Fig 3-6) though connected slightly differently. Connect the cable as follows:

1. Connect the gray Lemo connector on the Heat Capacity Controller Cable to the PPMS Probe Head.
2. Attach the large connector at the other end labeled “Heat Capacity Controller” to the CM-E Heat Capacity Module.
3. Attach the small 4-pin Lemo connector labeled “P1 User Bridge” and “P2 System Bridge” to the appropriate bridge connection. For Helium-3, connect it to the “P1-User Bridge” port on the Model 6000. For Standard Heat Capacity connect it to the “P2-System Bridge” port on the Model 6000.

If running Heat Capacity with the DR Insert, you will use the DR Heat Capacity Cable/RF Filter Box (Fig. 9-2). The DB-15 end of this cable is connected to the CM-E Heat Capacity Module. The end with the filter box will be connected to the DR probe head once the DR probe has been inserted in the PPMS sample chamber. Refer to Chapter 9 for more details.

2.3.3 Set Up the Sample-Mounting Station



WARNING !

Place the vacuum pump on the floor, not on a table next to the sample-mounting station. Because a vacuum hose connects the pump to the sample-mounting station, if the pump were to fall off the table, it would pull the sample-mounting station off the table.

1. Place the vacuum pump on the floor.
2. Verify that the power outlet you will plug the vacuum pump into has the correct voltage to operate the pump. Plug in the pump if the outlet has the correct voltage. Turn on the pump if it is equipped with a power switch.
3. Place the sample-mounting station on a table that is near the vacuum pump.
4. Connect the vacuum hose from the vacuum pump to the hose barb located at the rear of the sample-mounting station. Verify that the hose is securely connected to the hose barb.

2.4 Performing a First Addenda Measurement

Once the system is installed, you are ready to try a first measurement. This section is intended to introduce you to the basic operation of this system without a Helium-3 or DR insert. Rather than guide you through both the addenda measurement and the sample measurement, this section describes performing only an addenda measurement. The addenda measurement consists of measuring the heat capacity of the sample holder. This measurement is usually performed prior to mounting a sample for the purpose of separating the sample's heat capacity contribution from the total heat capacity.

1. Launch the MultiVu software on the computer and then activate the Heat Capacity option from the **Utilities>>Activate Option** menu command. When the Heat Capacity control center appears, click on the **Prepare Addenda Measurement** button. This launches a wizard to help you install the puck for an addenda measurement. See Chapter 4 for more details on starting the software.
2. You should have received a calibrated puck (see Figure 3-2) with your system. Apply a small amount of H Grease to the fingers, and then follow the instructions in the wizard to open the chamber, insert the puck, and purge the chamber. Be sure to note the serial number located on the puck since this is needed in the next part of the wizard. Also, make sure you are using the contact baffle assembly with the charcoal holder as shown in Figure 3-7. If you wish to perform a heat capacity measurement on a sample after completing this demonstration measurement, you should have applied some adhesive grease to the platform as described in Section 6.3.1.
3. Continue following the wizard, clicking **Next** when necessary. After entering the puck serial number, you will be asked to verify that the current calibration file is correct. The serial number shown must match the one you entered. To select the correct calibration file, you must click the **Change** button and select it from the list.
4. After you select the correct calibration file for the puck, the system will perform a test of the puck when you press the **Next** button. Do not continue until you get the "Success!" message. If the system fails to correctly verify the puck readings, check the cabling and press the **Test Again** button to retry.
5. In the next part of the wizard you will need to open a data file for saving the measurements. Select the **Open New File** button and specify the file. After defining the data file and returning to the wizard, click **Finish** to complete the installation.
6. Back in the **Measurement** tab of the Heat Capacity control center, click the **Create New Addenda Table** button to open the measurement dialog.
7. From the measurement dialog, click the **Suggest Defaults** button to fill in parameter suggestions. For a shorter run, try changing the **Start** and **End** temperatures to 300 and 200 with five temperature set points. A full description of the parameters is contained in Section 6.4.
8. Click **OK** to begin the measurement. The Measurement Status Viewer (Figure 4-4) then appears and displays the progress of each measurement.
9. After a few measurements, you can plot the data by first minimizing the Measurement Status Viewer, and then clicking the **Data File** button in the **View** section of the Heat Capacity control center. Minimize the control center so that it does not cover the graph. The addenda measurement continues to run even while minimized.
10. Once the measurement is complete, you may warm up and vent the chamber and remove the puck. If you intend to continue with a sample measurement, please refer to Chapter 7 for more details on repeating the above steps with a sample installed.

Hardware

3.1 Introduction

This chapter contains the following information:

- Section 3.2 discusses and illustrates the Heat Capacity hardware components.
- Section 3.3 discusses the contact baffle that is part of the High-Vacuum system.

3.2 Heat Capacity Hardware

This section describes the basic Heat Capacity option hardware. Please refer to Chapter 8 for Helium-3-specific option hardware, Chapter 9 for DR-specific hardware, or Chapter 10 for VersaLab-specific hardware or Chapter 11 for DynaCool-specific hardware.

3.2.1 Calorimeter Puck

The calorimeter puck (see Figure 3-1 on the following page) contains a resistive platform heater, platform thermometer, and puck thermometer. The platform heater and platform thermometer are attached to the bottom of the calorimeter chip that functions as the sample platform. The puck thermometer is buried within the puck. The platform thermometer measures the temperature of the sample platform and thus the temperature of the sample. The puck thermometer measures the temperature of the puck, which serves as the calorimeter's thermal bath.

Eight delicate, thermally conducting wires suspend the sample platform in the middle of the puck frame. These wires—four on each side of the platform—form the electrical connection to the platform heater and platform thermometer and also serve as the thermal connection between the platform and the puck frame. The puck frame and the wire guard help protect the wires, although the wires remain partially exposed.

The chuck, which is below the puck frame and above the green fiberglass connector, produces the thermal contact to the base of the sample chamber. The chuck includes the indexing key, which drops into the indexing notch to lock the puck in position inside the sample chamber.

Calibrated pucks may be purchased from Quantum Design. Unless a calibrated puck has been purchased, a puck is not calibrated when it is shipped from the factory. If a calibration file has not

been supplied with the puck, the puck must be calibrated before it may be used to measure heat capacity. Chapter 5 discusses puck calibration.

3.2.1.1 PUCK FRAME

The puck frame contains the thermally conducting wires and the sample platform. The puck has been designed so that the puck frame may be replaced if it is damaged. The Heat Capacity option includes a spare puck frame.

If a thermally conducting wire breaks, you remove and replace the puck frame. Two screws on top of the puck frame and two on the bottom attach the frame to the puck body. Section 9.4.1 explains how you remove and replace the puck frame.

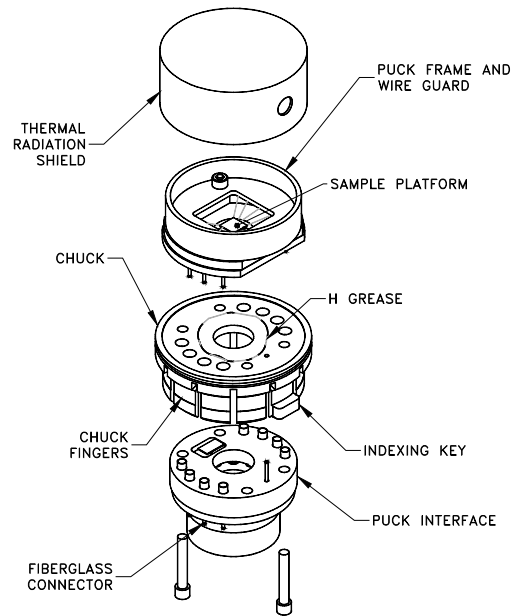


Figure 3-1. Exploded View of Calorimeter Puck

3.2.1.2 THERMAL RADIATION SHIELD

A thermal radiation shield fits snugly over the top of the puck. During a measurement, the shield protects the sample platform and the sample from unwanted heating that is created by warmer surfaces in the sample chamber, thereby guaranteeing a more accurate reading of the temperature of the sample.

The thermal radiation shield is also a protective cap that covers the thermally conducting wires and the sample platform. To prevent the wires and platform from being damaged, keep the thermal radiation shield on the puck when you are not using the puck for a measurement.

The rim of the shield is formed so it is not perfectly circular. Therefore, a snug fit between the shield and the puck frame is achieved by placing the shield onto the puck and twisting the cap until the fit is tight enough to easily support the weight of the assembly by only holding the lid. To remove the shield, untwist it to loosen. If the lid does not tighten properly, then you can carefully roll one edge of the shield on a hard surface to deform it slightly.

3.2.1.3 SERIAL NUMBER

At the factory, a unique serial number is assigned to each puck. The serial number distinguishes the puck and its associated calibration files from all other pucks used in the system. The serial number is written on the green fiberglass connector that is at the base of the puck. Refer to Figure 3-2.

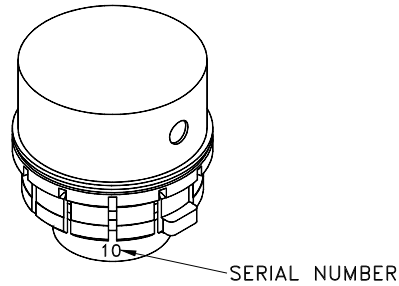


Figure 3-2. Location of Serial Number on Calorimeter Puck

3.2.2 Sample-Mounting Station

To prevent damage to the eight wires that hold the sample platform, the sample-mounting station uses a puck interlock arm and vacuum suction to stabilize the puck and platform. By stabilizing the puck and platform, the mounting station allows a sample to be easily mounted on the platform and removed from the platform while preventing the wires from being stressed.

The puck interlock arm is curved to fit the circular exterior of the puck holder. When the interlock arm is pushed toward the holder, the interlock arm clasps and helps immobilize the puck. The vacuum supplied at the hose barb appears at the hole in the platform holder and sucks the platform downward to hold it in place. Small fingers on top of the platform holder further stabilize the platform.

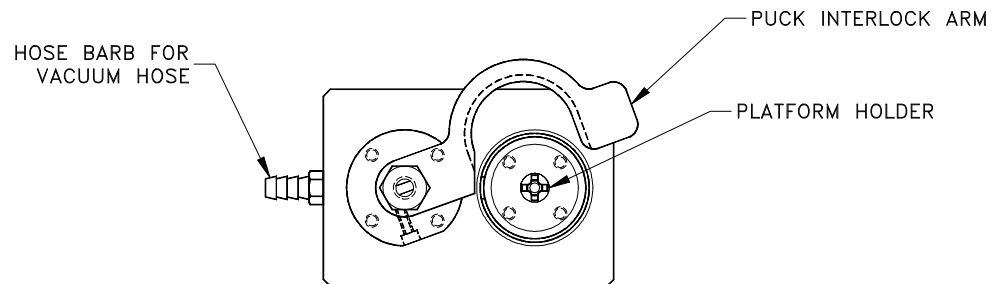


Figure 3-3. Top View of Sample-Mounting Station

3.2.2.1 VACUUM PUMP

The vacuum pump is part of the sample-mounting station assembly. Vacuum is applied through the vacuum hose that attaches to the hose barb at the rear of the sample-mounting station.



WARNING!

Place the pump on the floor or securely mount it to a stable surface. The pump vibrates when it operates and can “walk” off the edge of a table, dragging any attached object—including the sample-mounting station—with it.

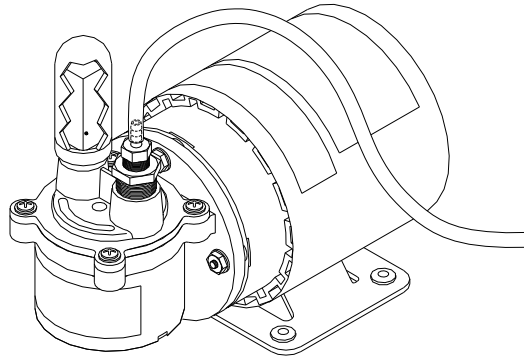


Figure 3-4. Vacuum Pump for Sample-Mounting Station

3.2.3 Model CM-E Heat Capacity Control Module

The Model CM-E Heat Capacity Module contains the electronics that simultaneously control the heat applied by the platform heater and measure the temperature of the platform thermometer. (The platform heater and platform thermometer are on the calorimeter puck. Refer to Section 3.2.1.) A dedicate processor and its quartz crystal clock provide the precise timing of the heater drive and thermometry that is critical to accurate heat capacity measurements.

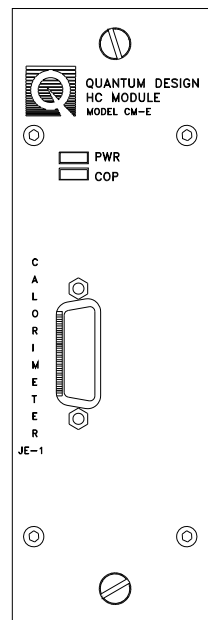


Figure 3.5 Heat Capacity CAN Module

A digital-to-analog converter and a precision current source apply power to the platform heater. The programmable current ranges allow precision currents ranging from a few nanoamps to a few milliamps to be driven in the heater. An analog-to-digital converter (ADC) and a programmable gain stage monitor the heater power. This arrangement, combined with the current source used for driving the heater, allows precision determination of the applied heater power as a function of time.

A precision current source provides the AC excitation for reading the platform thermometer while a high-precision, high-accuracy ADC reads the voltage across the thermometer. The resistance of the thermometer is measured by driving a square-wave alternating current at 250 Hz in the thermometer. Using a high-precision, high-accuracy ADC, the embedded processor measures the voltage across the thermometer during both the positive and negative part of the 250-Hz cycle. The difference between the two readings provides a differential measurement of the resistance at 4-ms intervals. Onboard calibration resistors with low-temperature coefficient are used for absolute accuracy in the resistance measurement.

This module was designed for the special requirements of performing heat capacity measurements at Helium-3 and DR temperatures down to 50 mK (low excitations and offset currents) and also the higher power requirements of standard heat capacity measurements up to 400 K. The Model CM-E module can be used for all versions of the Heat Capacity Option.

During a typical measurement, the following occurs: The parameters for a single heater-on–heater-off cycle are loaded into the module’s local memory. When the parameters are loaded, the measurement is triggered, and the processor in the module sets the heater current and records the thermometer resistance at 4-ms intervals. At the same time the thermometer is read, the heater voltage is recorded and stored for use in computing heater power. When the measurement is complete, the thermometer readings and the heater voltages are read out of the module’s local memory via the CAN bus and the results processed by the Heat Capacity software running on the PC.

3.2.4 Controller Cable Assembly

The Heat Capacity controller cable assembly carries the thermometer and heater excitation signals between the Model CM-E Heat Capacity controller and the calorimeter puck for both the helium-3 and standard heat capacity operation. It also connects the auxiliary puck thermometer (Standard Option) or the main temperature-control thermometer (Helium-3 Option) to a bridge channel in the Model 6000. See Section 2.3.2.3 for specific instructions on connecting this cable.

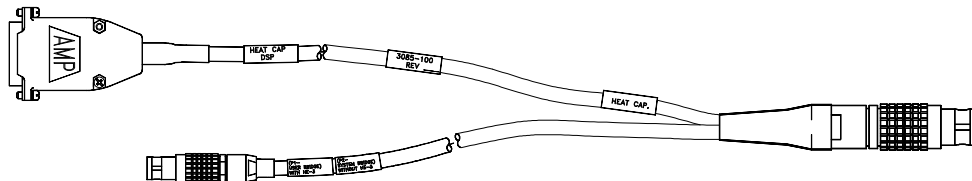
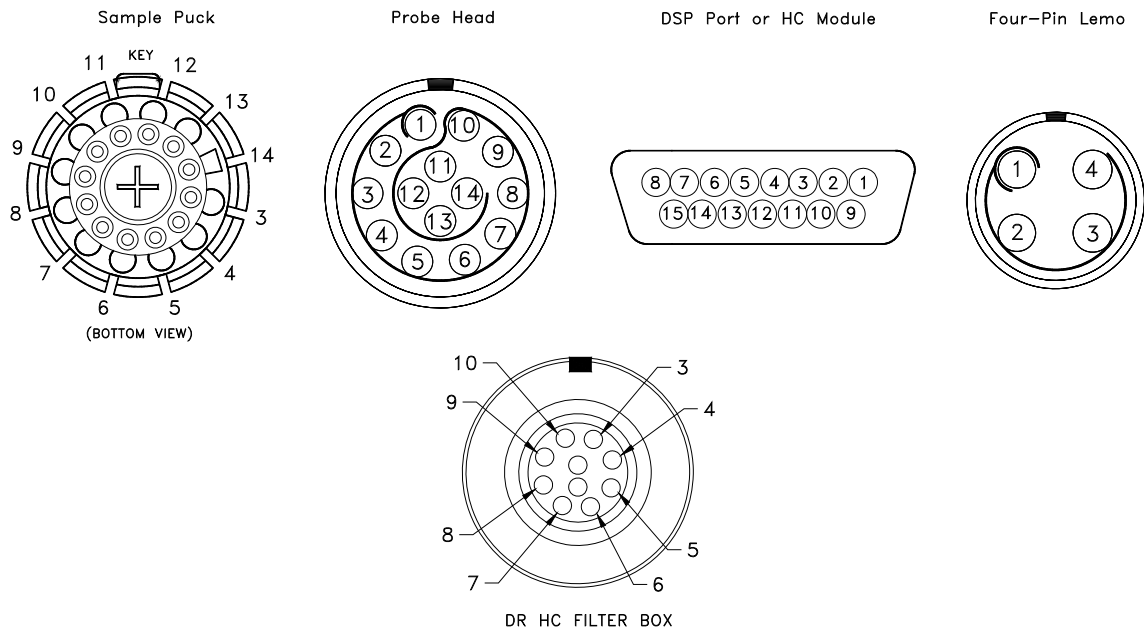


Figure 3-6 Controller Cable

A separate cable assembly having an integrated RF filter box (see Figure 9-2) is used with the DR option.

Table 3-1. Sample Connections for Pin Numbers



PUCK	GRAY LEMO CONNECTOR AT PROBE HEAD	4-PIN LEMO ON MODEL 6000 AT BRIDGE CHANNEL 4	DSP PORT OR HC MODULE	DR HC FILTER BOX	DESCRIPTION
3	3		3	3	Heater I+
4	4		10	4	Heater I-
5	5		4	5	Heater V+
6	6		11	6	Heater V-
7	7		2	7	Chip Therm I+
8	8		9	8	Chip Therm I-
9	9		8	9	Chip Therm V+
10	10		15	10	Chip Therm V-
11	11	1			Puck Therm I+
12	12	2			Puck Therm I-
13	13	3			Puck Therm V+
14	14	4			Puck Therm V-
			1		Ground

3.3 High-Vacuum Hardware

The High-Vacuum system, which operates in conjunction with the Heat Capacity option, reduces the amount of gas in the sample chamber and thus minimizes the paths by which heat escapes the sample platform. The Heat Capacity option works with either the Turbo Pump (found on some PPMS systems) or Cryopump High-Vacuum systems.

3.3.1 Contact Baffle for Standard Heat Capacity

An integral part of either the Turbo Pump or the Cryopump High-Vacuum system is the contact baffle assembly. The contact baffle makes thermal contact with the isothermal region of the sample chamber, which is just above the puck. The thermal contact between the contact baffle and the isothermal region helps create a more uniform thermal environment for the Standard Heat Capacity puck by causing the contact baffle to be at the same temperature as the chamber walls that are near the puck. This is important when high vacuum is enabled; high vacuum reduces the amount of thermal exchange gas in the sample chamber.

You screw the contact baffle into the brass fitting that is at the bottom of the baffle assembly (Figure 3-7). To reduce unnecessary wear on the contact baffle, use it only when you are using the High-Vacuum system. Handle the contact baffle with care, and avoid touching the delicate outer contact fingers.

The contact baffle includes a removable charcoal holder that screws into the bottom of the baffle assembly (Figure 3-8). The charcoal holder helps prevent helium from adsorbing on the sample platform when the temperature of the sample chamber is below 10 K. It is removed only for puck calibrations.

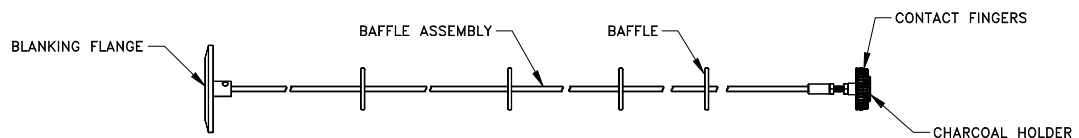
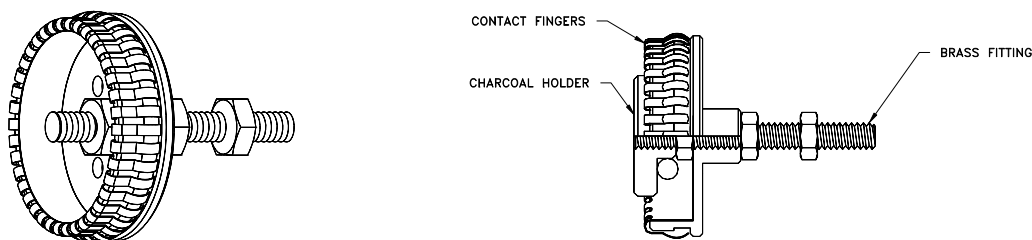


Figure 3-7. Baffle Assembly with Contact Baffle



A. Charcoal Holder Removed for Puck Calibration

B. Charcoal Holder Installed for Normal Operation

Figure 3-8. Close-up View of Contact Fingers and Charcoal Holder on Contact Baffle Assembly

Software

4.1 Introduction

This chapter contains the following information:

- Section 4.2 presents an overview of the Heat Capacity software.
- Section 4.3 discusses the analysis models used for heat capacity measurements.
- Section 4.4 discusses the Heat Capacity control center.
- Section 4.5 discusses the Measurement Status Viewer and measurement fields.
- Section 4.6 discusses the Raw Data Browser and Slope Analysis.
- Section 4.7 presents an overview of the Heat Capacity sequence commands.
- Section 4.8 discusses the Heat Capacity data files.
- Section 4.9 discusses message and error log files.

4.2 Overview of System Software

The Heat Capacity software application, which is the software for the Heat Capacity option, handles all aspects—from the low-level control of heaters and thermometers to the final conversion to heat capacity numbers and subtraction of addenda—of the calorimeter operation. The Heat Capacity software also manages the calibration files and saves calibration or measurement data to any open data file. The Heat Capacity software is activated from within MultiVu by selecting the **Utilities>>Activate Option** menu command.

Heat Capacity measurements may be performed either directly from the Heat Capacity control center or from sequence commands. All measurement data is written to the current Heat Capacity data file and associated raw data file, as defined in the control center. The calibration information for a specific calorimeter puck is contained in the currently defined calibration file. The data file, calibration file, and currently defined addenda table are identified in the Heat Capacity control center (see Figure 4-1, for example). Installation wizards guide you through the process of installing and testing the calorimeter puck as well as defining the necessary files prior to running a measurement.

Table 4-1. Software Files for Heat Capacity Option

Directory (under \QdPpms, \QdVersaLab, or \QdDynaCool)	Files	Description	Manual Reference
\Heat Capacity\System	MvHeatCap.dll HeatCap.hwc HeatCap.chm HeatCapStandard.Ini HeatCapHe3.Ini HeatCapDR.Ini HeatCapVersaLab.Ini	Executable DSP board calibration Help file Configuration for “Heat Capacity” option Configuration for “Heat Capacity for He3” option Configuration for “Heat Capacity for DR” option Configuration for VersaLab Heat Capacity Option	
\Heat Capacity\TempCal	*.off	Addenda offset tables	Section 6.7.3
\Heat Capacity\TempCal\Standard	*.cal Standard.pkd	Puck calibration and puck-type description files for “Heat Capacity” option	Section 5.2.2 Section 5.2.1
\Heat Capacity\TempCal\He3	*.cal He3Puck.pkd	Puck calibration and puck-type files for “Heat Capacity for He3” option	
\Heat Capacity\TempCal\DR	*.cal DRPuck.pkd	Puck calibration and puck-type files for “Heat Capacity for DR” option	
\Heat Capacity\TempCal\VersaLab	*.cal VLPuck.pkd	Puck calibration and puck-type files for VersaLab Heat Capacity Option	
\Heat Capacity\LogFiles	Errors.txt Messages.txt *.raw, *.fit (obsolete)	Text Error Messages Messages from Measurement Status Viewer Obsolete raw and fit files. Use the .raw file described in Sections 4.6 and 4.8.4 instead.	Section 4.9 Section 4.9

4.3 Analysis Models

4.3.1 Thermal Models

Central to the conversion of raw data into heat capacity numbers is the mathematical model used to describe the temperature response of the sample platform as a function of time.

4.3.1.1 SIMPLE MODEL

The simple model, which is the most basic analysis of the raw measurement data, assumes that the sample and sample platform are in good thermal contact with each other and are at the same temperature during the measurement. In the simple model, the temperature T of the platform as a function of time t obeys the equation

$$C_{\text{total}} \frac{dT}{dt} = -K_w(T - T_b) + P(t), \quad (4.1)$$

where C_{total} is the total heat capacity of the sample and sample platform; K_w is the thermal conductance of the supporting wires; T_b is the temperature of the thermal bath (puck frame); and $P(t)$ is the power applied by the heater. The heater power $P(t)$ is equal to P_0 during the heating portion of the measurement and equal to zero during the cooling portion. The solution of this equation is given by exponential functions with a characteristic time constant τ equal to C_{total}/K .

The Heat Capacity software uses the simple model to measure the addenda and generally uses the simple model to measure most samples. However, when the thermal contact between the sample and sample platform is poor, the software uses the more sophisticated two-tau model to measure the heat capacity.¹

4.3.1.2 TWO-TAU MODEL

The Heat Capacity software uses the two-tau model to measure the heat capacity of the sample when poor thermal attachment of the sample to the platform produces a temperature difference between the two. The two-tau model simulates the effect of heat flowing between the sample platform and sample, and the effect of heat flowing between the sample platform and puck. The following equations express the two-tau model:

$$C_{\text{platform}} \frac{dT_p}{dt} = P(t) - K_w(T_p(t) - T_b) + K_g(T_s(t) - T_p(t)) \quad (4.2)$$

$$C_{\text{sample}} \frac{dT_s}{dt} = -K_g(T_s(t) - T_p(t)),$$

where C_{platform} is the heat capacity of the sample platform, C_{sample} is the heat capacity of the sample, and K_g is the thermal conductance between the two due to the grease. The respective temperatures of the platform and sample are given by $T_p(t)$ and $T_s(t)$.

¹ Two-tau model™ is a trademark of Quantum Design.

4.3.2 Data Fitting

Using a nonlinear, least-square fitting algorithm, the system compares the solution to the simple model to the actual measurement. The values of the parameter that give the smallest fit deviation determine the heat capacity. The sensitivity of the fit deviation (chi square) to small variations in the fitting parameters is used to estimate the standard errors for the heat capacity.

Except when measuring the addenda (the heat capacity of only the platform), a fit to the solution of the two-tau model is also performed. The heat capacity numbers derived in this way are used only if the fit has a smaller fit deviation than in the first case. Under some circumstances, the fit to the two-tau model does not converge, in which case the simple fit is again used. Such a lack of convergence can occur when the sample is perfectly attached to the platform, in which case the simple model is correct. Fitting difficulties can also arise in other cases where the sample is poorly attached and the heat capacity of the sample is not large compared to the platform. In either case, the sample coupling is shown to be 100%.

4.3.3 Derived Quantities from Model Parameters

4.3.3.1 SIMPLE MODEL: ADDENDA MEASUREMENT

An addenda measurement uses only the simple model (Section 4.3.1.1) for computing heat capacity. In this case, the measurement fields in the Measurement Status Viewer (Section 4.5) are computed as indicated in table 4-2.

Table 4-2. Simple Model Parameters Used for Addenda Measurement

STATUS VIEWER FIELD	EXPRESSION FROM MODEL
Total Heat Cap	C_{total}
Sample Heat Cap	zero
Addenda Heat Cap	C_{total}
Time Const (tau1)	C_{total} / K_w
Time Const (tau2)	Zero
Sample Coupling	100%

4.3.3.2 SIMPLE MODEL: SAMPLE MEASUREMENT

When the analysis of a sample heat capacity measurement fails to find a fit to a two-tau solution, the simple fit (Section 4.3.1.1) is used as in the case of the addenda measurement. The Sample Heat Cap measurement field is computed by subtracting the total heat capacity of the fit from the addenda heat capacity C_{addenda} as retrieved from a previously measured addenda table. In this case, the measurement fields in the Measurement Status Viewer (Section 4.5) are computed as indicated in table 4-3.

Table 4-3. Simple Model Parameters Used for Sample Heat Capacity Measurement

STATUS VIEWER FIELD	EXPRESSION FROM MODEL
Total Heat Cap	C_{total}
Sample Heat Cap	$C_{\text{total}} - C_{\text{addenda}}$
Addenda Heat Cap	C_{addenda}
Time Const (tau1)	C_{total}/K_w
Time Const (tau2)	Zero
Sample Coupling	100%

4.3.3.3 TWO-TAU MODEL: SAMPLE MEASUREMENT

When the two-tau model (Section 4.3.1.2) is fit to the measurement data for a sample heat capacity measurement, the addenda heat capacity C_{addenda} is retrieved from a previously measured addenda table and C_{platform} is treated as a constant equal to C_{addenda} in the fitting routine. In this case, the measurement fields in the Measurement Status Viewer (Section 4.5) are computed as indicated in table 4-4.

Table 4-4. Two-Tau Model Parameters Used for Sample Heat Capacity Measurement

STATUS VIEWER FIELD	EXPRESSION FROM MODEL
Total Heat Cap	$C_{\text{platform}} + C_{\text{sample}}$
Sample Heat Cap	C_{sample}
Addenda Heat Cap	C_{platform}
Time Const (tau1)	$1/(\alpha - \beta)$
Time Const (tau2)	$1/(\alpha + \beta)$
Sample Coupling	$100 \times K_g/(K_g + K_w)$

The expressions for α and β are given by

$$\alpha = \frac{K_w}{2C_{\text{platform}}} + \frac{K_g}{2C_{\text{platform}}} + \frac{K_g}{2C_{\text{sample}}},$$

$$\beta = \frac{\sqrt{K_g^2 C_{\text{sample}}^2 + 2K_g^2 C_{\text{sample}} C_{\text{platform}} + K_g^2 C_{\text{platform}}^2 + K_w^2 C_{\text{sample}}^2 + 2K_w C_{\text{sample}}^2 K_g - 2K_w C_{\text{sample}} K_g C_{\text{platform}}}}{2C_{\text{platform}} C_{\text{sample}}}. \quad (4.3)$$

4.3.3.4 EQUIVALENT DEBYE TEMPERATURE

The system can also express sample heat capacity as an equivalent Debye temperature in the event that the sample mass, formula weight, and atoms per formula unit have been entered.

The Debye model successfully describes the heat capacity of phonons (lattice contribution). Phonons, electrons, and magnons all contribute to the heat capacity in solids.

The general expression for lattice heat capacity and temperature T can be stated as

$$C_v = 3rNk \int_0^{\infty} \left(\frac{h\nu}{2kT} \right)^2 \csc^2 \left(\frac{h\nu}{2kT} \right) g(\nu) d\nu, \quad (4.4)$$

where r is the number of atoms per molecule, N is the number of molecules, and k is the Boltzmann constant. The density of phonon modes in the frequency range from ν to $\nu + d\nu$ is given by $g(\nu)d\nu$. In the Debye model, the mode density function $g(\nu)$ is derived by assuming that phonon propagation through the crystal lattice is governed by the same dispersion relation as linear waves in a continuous isotropic solid. To account for the lattice spacing in a real solid, Debye's theory specifies a cutoff frequency, ν_D , above which the mode density function is zero. That is,

$$g(\nu) = \frac{3\nu^2}{\nu_D^3} \text{ for } \nu \leq \nu_D$$

$$g(\nu) = 0 \text{ for } \nu > \nu_D. \quad (4.5)$$

Physically, ν_D corresponds to the smallest phonon wavelength that can propagate in a lattice of atoms with a finite spacing. It is related to both the speed of sound and the elastic properties of the solid. The Debye temperature is then defined as θ , where $h\nu_D = \theta k$. Putting $g(\nu)$ into the above expression for heat capacity creates the following relation between C_v and θ :

$$C_v = 9rNk \frac{T^3}{\theta^3} \int_0^{\theta/T} \frac{x^4 e^x dx}{(e^x - 1)^2}. \quad (4.6)$$

This expression contains the two well-known limits of heat capacity for nonmetallic solids. At high temperatures, $C_v \rightarrow 3rNk$, whereas at sufficiently low temperatures,

$$C_v \longrightarrow \frac{12}{5} rNk \pi^4 \left(\frac{T}{\theta} \right)^3,$$

which is the familiar T^3 law.

Experimentally, you can determine θ by first measuring the heat capacity of a known quantity N of molecules, each molecule having r atoms, at a temperature T . You then numerically solve the above expression for θ . For a perfect Debye solid, θ does not change with temperature. However, in real solids, lattice effects and electronic and other contributions to the heat capacity cause θ to vary with temperature.

It is important to point out that the value for Debye temperature which is computed by the application assumes that the sample heat capacity contains only a lattice contribution. No special accounting is made for electronic, magnetic, or structural terms.

Plots of equivalent Debye temperature as a function of temperature were first suggested by M. Blackman in "The Theory of the Specific Heat of Solids" (Blackman 1942).

4.3.3.5 MEASUREMENT UNITS

Table 4-5 summarizes common measurement units that express heat capacity and specific heat. The default measurement units, $\mu\text{J/K}$, express heat capacity. The sample information that is defined in the open data file determines which measurement units, other than $\mu\text{J/K}$, are available. You define sample information in the header of the data file you have opened to log the measurement. If you neglect to define all sample information, the application still collects data, but it expresses the data in the units for heat capacity, $\mu\text{J/K}$, rather than in the units you have selected.

Table 4-5. Common Units for Heat Capacity and Specific Heat

	UNITS	FORMULA
HEAT CAPACITY	$\mu\text{J/K}$	
MASS SPECIFIC HEAT	$\mu\text{J/mg-K}$	$\mu\text{J/K/mass}$
	$\mu\text{J/g-K}$	$\mu\text{J/K/mass}$
	J/g-K	$0.001 \times (\mu\text{J/K})/\text{mass}$
	cal/g-K	$0.0002390057 \times (\mu\text{J/K})/\text{mass}$
MOLAR SPECIFIC HEAT	mJ/mole-K	$(\mu\text{J/K}) \times \text{molwght}/\text{mass}$
	J/mole-K	$0.001 \times (\mu\text{J/K}) \times \text{molwght}/\text{mass}$
	cal/mole-K	$0.0002390057 \times (\mu\text{J/K}) \times \text{molwght}/\text{mass}$
GRAM-ATOM SPECIFIC HEAT	J/gat-K	$0.001 \times (\mu\text{J/K}) \times \text{molwght}/(\text{mass} \times \text{atoms})$
	cal/gat-K	$0.0002390057 \times (\mu\text{J/K}) \times \text{molwght}/(\text{mass} \times \text{atoms})$

4.3.3.6 ESTIMATED HEAT CAPACITY ERROR

In both the data file (see Table 4-10) and the Measurement Status Viewer (Figure 4-4), the heat capacity is given with an estimated error term. The software calculates this error based on the fit and the entered sample mass properties.

There are four terms in the expression for sample heat capacity error:

1. Total Heat Capacity Error. This error comes from calculating the sensitivity of the mean-square deviation of the fit to variations in the heat capacity parameter. Recall that the heat capacity is a parameter in the fit.
2. Addenda Heat Capacity Error. This is calculated during the addenda measurement and is saved in the addenda table for this purpose.

3. Sample Mass Error. If a mass error term is entered with the sample information when the data file is created, and the results are expressed as a heat capacity per unit mass, then the fractional mass error is included.
4. Fit Deviation Error. To take into account modeling errors where there is a large deviation between the fit and the measurement, yet the measurement noise is small, the software includes this term, equal to the fit deviation (in Kelvin) divided by the temperature rise. Note that this last term is not a random error, but rather a modeling error.

All four of these terms are added in quadrature to obtain an estimate of the error bars for a measurement.

4.3.4 Slope Analysis of Relaxation Curves

The thermal models and the data-fitting algorithm described in sections 4.3.1 and 4.3.2 assume that the heat capacity is approximately constant over the range of temperatures covered by a single measurement cycle. However, this assumption is not always true.

4.3.4.1 SINGLE-CURVE SLOPE ANALYSIS (FIRST-ORDER TRANSITIONS)

In first-order transitions, for example when there is a “latent heat” associated with a transition, the heat capacity behaves as if it is infinite at the transition temperature. In real samples, such a transition appears as a very narrow peak in the heat capacity. If the width of this peak is smaller than the temperature rise of the relaxation measurement, the result will be a considerable smearing of the peak with the possibility that it will be completely missed.

When the sample temperature is warmed and cooled through a first-order transition, the resulting temperature response curve will contain information about the temperature dependence of the heat capacity. The central equation for this analysis is found by rearranging the Simple Model from section 4.3.1.1 to express C_{total} as a function of temperature:

$$C_{\text{total}}(T) = \frac{-K_w(T - T_b) + P(T)}{S(T)}, \quad (4.7a)$$

where $S(T) = dT/dt$ is the slope of the relaxation curve expressed as a function of temperature and $P(T)$ is the heater power as a function of temperature.

During a single relaxation measurement, both the heating and cooling portions of the curve can be used to obtain heat capacity using this expression. The heater power $P(T)$ is a constant P_0 during the heating portion of the curve and zero during the cooling portion. This yields two $C_{\text{total}}(T)$ curves for each relaxation measurement. The thermal conductance K_w of the wires can be approximated using the standard curve-fitting method described earlier.

In practice, the temperature dependence of $K_w(T)$ comes from the wire conductance table created during pass 2 of the calibration procedure as described in section 5.6.2. The software implementation of the single-curve slope analysis also takes this temperature dependence into account and additionally includes an *ad hoc* offset parameter $K_{\text{os}} = K_w(T_b) \times (\text{static offset}) / 100\%$ (see **static offset** in Fig. 4-5). Therefore, a more accurate description of the single-curve slope analysis algorithm used by the software is given by

$$C_{\text{total}}(T) = \frac{-\int_{T_b}^T (K_w(T') + K_{os})dT' + P(T)}{S(T)}. \quad (4.7b)$$

The heating (or cooling) rate $S(T)$ must be estimated from a potentially noisy set of temperature measurements. Therefore, it is necessary to specify an averaging parameter to optimize temperature resolution versus heat capacity resolution. The software uses a slope averaging technique that is specified as a percentage of the duration of the heating curve (or cooling curve). For example, if this Moving Average Width (MAW) is given as 5%, then the slope $S(T)$ at a particular temperature is evaluated by fitting a line through the 5% of temperature measurements in the neighborhood of that temperature. Selecting MAW = 0 simply uses adjacent values of $T(t)$ in the measurement data, which is the minimum number of measurements needed to compute slope.

4.3.4.2 DUAL-SLOPE ANALYSIS (LARGE TEMPERATURE RISES)

The expression (4.7b) for C_{total} in the previous section is used to evaluate heat capacity as a function of temperature for both the heating and cooling segments of a relaxation curve. Being able to separate the heating curve from the cooling curve is particularly useful for analyzing relaxation data that crosses a first-order transition. This is because the sharp feature may appear slightly shifted in temperature depending on the temperature sweep direction. However, for samples without such sharp features, we can combine the separate heating and cooling curve solutions from the previous section into a single expression for C_{total} with a much simpler form (Riegel and Weber 1986):

$$C_{\text{total}}(T) = \frac{P_h(T) - P_c(T)}{S_h(T) - S_c(T)}. \quad (4.8)$$

In this expression, the “h” and “c” subscripts refer to the heating and cooling curves, respectively. It is derived by writing (4.7b) for both the heating and cooling curves then solving them simultaneously for $C_{\text{total}}(T)$, thus eliminating the thermal conductance term containing K_w . This has the desirable result of removing uncertainties introduced by relatively poor knowledge of the bath temperature T_b and the wire conductance K_w . Because this expression does not have explicit dependence on heat loss through the wires, it is possible to obtain accurate heat capacity values from relaxation curves that span 30% or more in temperature instead of the more typical 1-2%. This allows much wider temperature spacing in measurement sequences without compromising temperature coverage.

4.3.4.3 OPTIMIZING MEASUREMENTS FOR SLOPE-ANALYSIS

While it is possible to perform slope analysis using the above methods on any heat capacity data, to get the best results, care is needed in selecting measurement parameters. Because the dual-slope solution uses only the portions of the heating and cooling curve that overlap in temperature, it may be useful to adjust measurement parameters to maximize the overlap region. For example, increasing the “Measurement Time” parameter (See Chapter 7) to 3 time constants would increase the overlap to more than 50%. Then the temperature spacing could be selected with about 50% overlap to ensure that there are no temperature-gaps in the data. For example, if a 30% temperature rise is specified with log temperature spacing between temperatures T_{\min} and T_{\max} , then the temperature spacing ratio between neighboring temperatures is given by $R = (T_{\max} / T_{\min})^{1/(N-1)}$. You would then select the number of temperature values N so that the spacing ratio is less than about 1.15 (50% of a 30% spacing).

Another important consideration when using this analysis technique is the fact that it is based on the simple thermal model given by equation 4.1. If the sample exhibits a significant second time-constant, then the absolute accuracy of the results of this slope technique are expected to be worse than the standard two-tau fit would give. Hence, it may be necessary to obtain measurements using both techniques (large temperature rises with slope analysis and also small temperature rises with two-tau analysis) to verify accuracy.

Performing slope analysis using the Raw Data Browser is described in section 4.6.

4.4 Heat Capacity Control Center

The Heat Capacity software has a control center that includes all frequently selected Heat Capacity commands. With its easy-to-use tab format and software prompts, the Heat Capacity control center makes basic system operations, such as installing samples, creating data files or calibration files, and setting up and running immediate-mode measurements, more natural and convenient. The Heat Capacity control center opens as soon as the Heat Capacity option is activated, and although it may be minimized, does not close until the option is deactivated. Figures 4-1 through 4-3 illustrate the tabs in the Heat Capacity control center.

Command buttons in the **View** area that is below the tab portion of the Heat Capacity control center open the Measurement Status Viewer, the Heat Capacity error log, the header of the active data file, and a MultiVu graph of the current data file. The **Meas Status** button is always enabled. The **Error Log** button is enabled only when error messages have been logged to the Heat Capacity error log. The **Sample Info** button and the **Data File** button are enabled only when a data file is active.

The status area at the bottom of the Heat Capacity control center identifies the active calibration file and active addenda table and summarizes the general status of the Heat Capacity system.

4.4.1 Installation Wizards Tab

Commands in the **Installation Wizards** tab initiate the puck installation wizards. There are three puck installation wizards: one for calibration, one for addenda measurement, and one for sample measurement. Each wizard guides you through the different steps you perform to install a puck and prepare for a calibration or for an addenda or sample measurement. Using the puck installation wizards helps ensure that you perform all necessary procedures before you initiate a calibration or measurement.

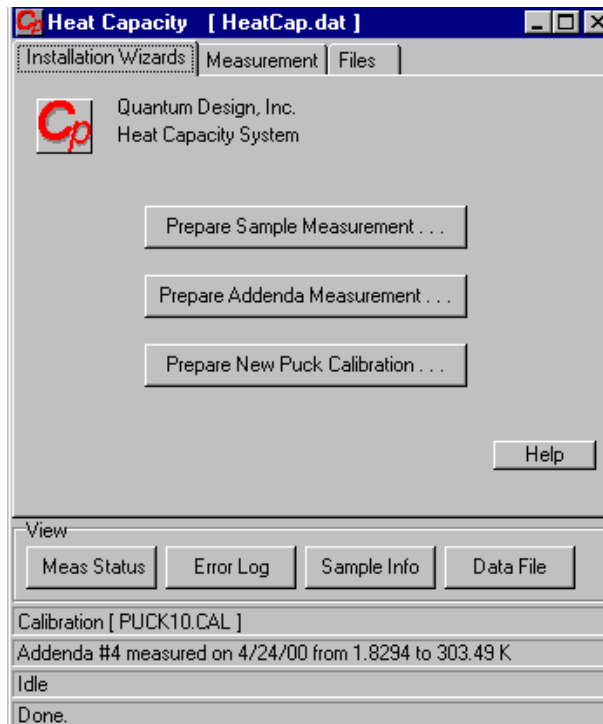


Figure 4-1. **Installation Wizards** Tab in Heat Capacity Control Center

4.4.2 Measurement Tab

Commands in the **Measurement** tab initiate sample or addenda measurements or calibrate the thermometer in a non-zero magnetic field.

Only one immediate-mode **Measurement** command may run at a time. All command buttons in the **Measurement** tab are disabled while a measurement runs.

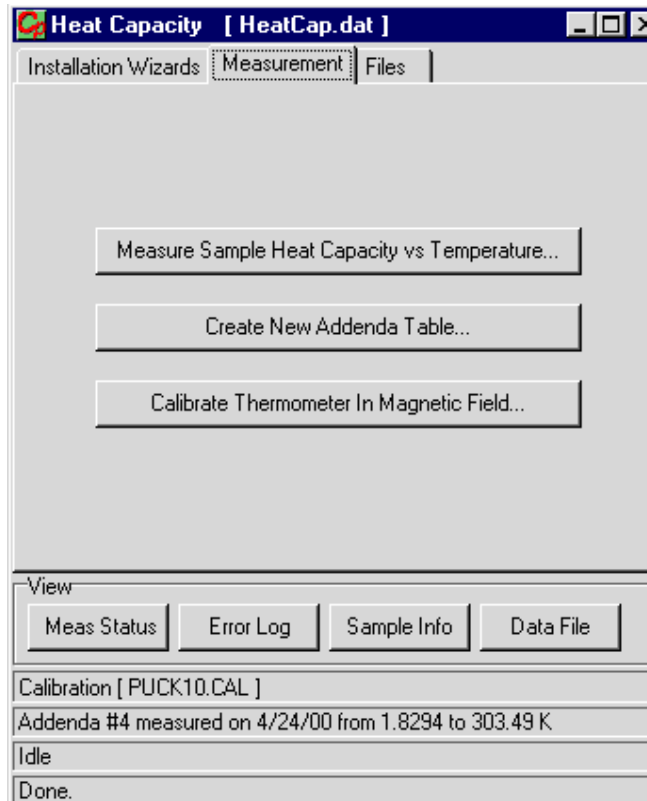


Figure 4-2. **Measurement** Tab in Heat Capacity Control Center

4.4.3 Files Tab

Commands in the **Files** tab select data files, calibration files, and addenda tables. The **Output Data File** commands create new data files, append measurement data to an existing data file, or close the active data file. The **Calibration File** commands select calibration files or addenda tables. The **Read and Reprocess** command, which is enabled only when a data file is active, reads raw heat capacity values from an inactive data file and writes each measurement to the active file. **Read and Reprocess** is normally used to repair a data file containing inaccurate or incomplete sample information.

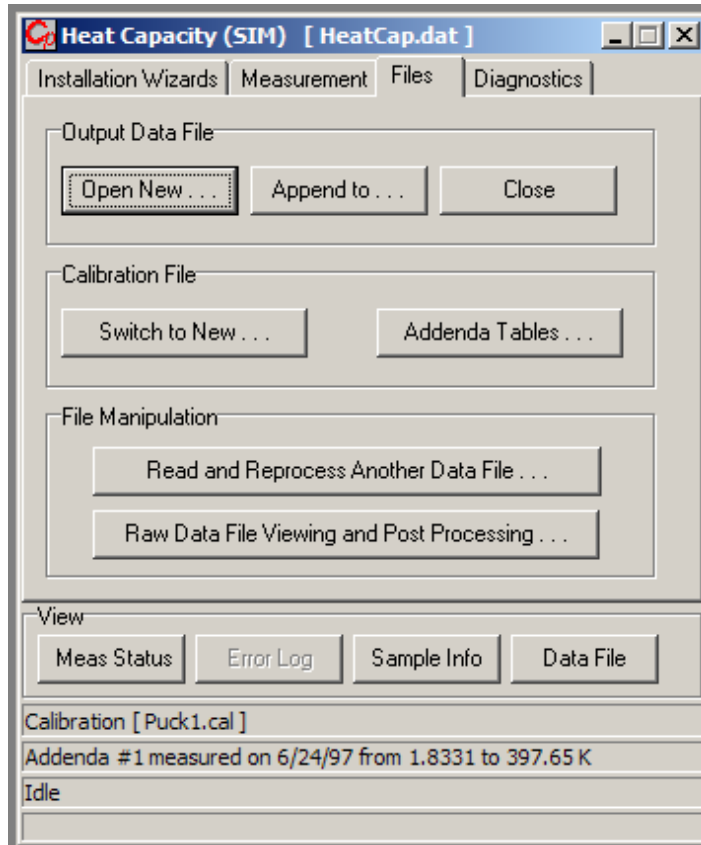


Figure 4-3. Files Tab in Heat Capacity Control Center

4.5 Measurement Status Viewer

As soon as a heat capacity measurement begins, the Measurement Status Viewer opens and indicates the progress of the measurement. The name of each task that is part of the measurement appears, as it is performed, in the message list box at the bottom of the Viewer. When the Heat Capacity software acquires data, it presents the data in the measurement-field panels, which are on the left side of the Viewer, and plots the raw data as a graph. Refer to Figure 4-4.

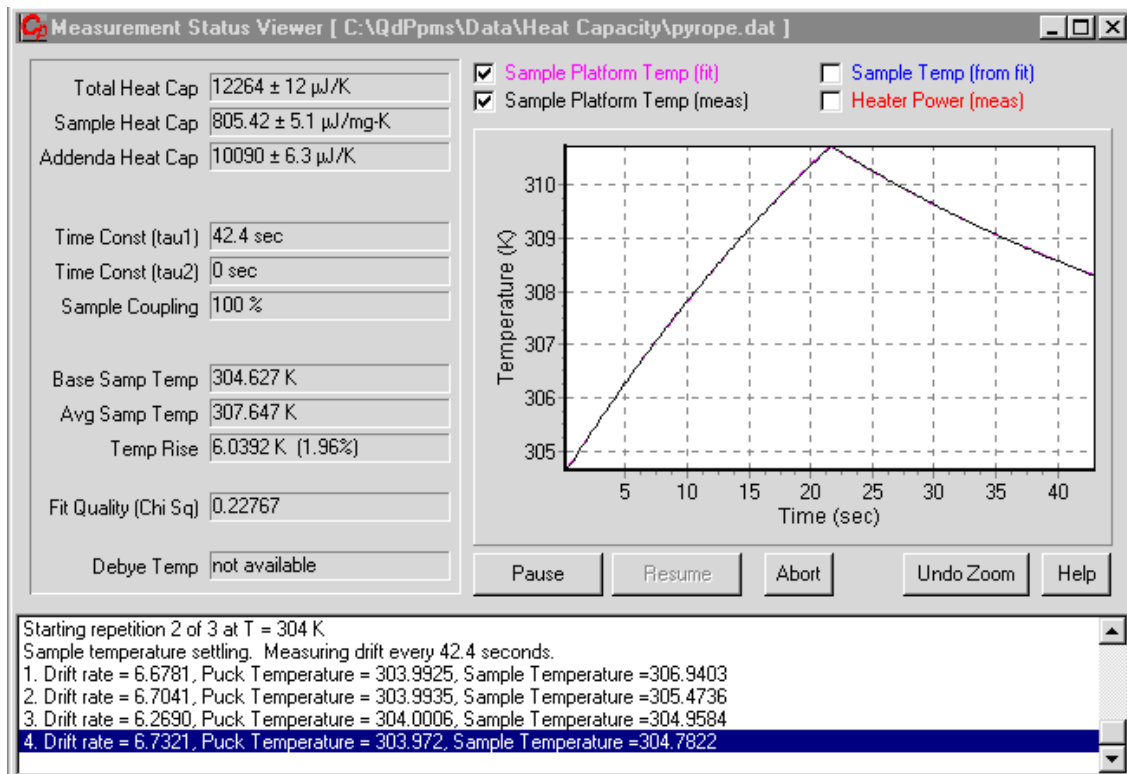


Figure 4-4. Measurement Status Viewer. The graph on the right side of the Viewer displays a plot of fit and measured temperature versus time.

4.5.1 Measurement Fields

To compute the value of most measurement fields that appear in the Measurement Status Viewer, the Heat Capacity software compares the measured temperature response of the sample platform with a theoretical model of the temperature response of the platform. By systematically varying the parameters in the theoretical model, the software determines the choice of parameters that provide the best fit. The *best fit* corresponds to a global minimum of the square deviation of the model from the measurement. In some cases, the software computes an error term, which is preceded by a plus and minus sign (\pm) in the measurement-field panel. The errors indicate to what extent the fit quality changes if the parameter changes.

Table 4-6. Measurement Fields in Measurement Status Viewer

MEASUREMENT FIELD	DEFINITION
Total Heat Cap	Total heat capacity, in $\mu\text{J/K}$, of sample and platform—if sample is on platform. Total Heat Cap is computed from fit.
Sample Heat Cap	Heat capacity of sample. Sample Heat Cap is computed from fit and addenda table. Value of field expresses heat capacity or specific heat, based on user-selected measurement units. For addenda measurement, Sample Heat Cap is zero. For sample measurement, Sample Heat Cap is computed by subtracting addenda, found in active addenda table, from Total Heat Cap. If addenda has not been measured, addenda value is zero, and only Total Heat Cap is given.
Addenda Heat Cap	Total heat capacity, in $\mu\text{J/K}$, of addenda. Addenda Heat Cap is computed from active addenda table by interpolating table values at average sample temperature for current measurement. For addenda measurement, Addenda Heat Cap is identical to Total Heat Cap. For sample measurement, Addenda Heat Cap is computed from active addenda table.
Time Const (tau1)	Long time constant, in seconds, of sample. For addenda measurement, Time Const (tau1) is single time constant from simple model. For sample measurement, Time Const (tau1) is tau1 from two-tau model.
Time Const (tau2)	Short time constant, in seconds, of sample. For addenda measurement, Time Const (tau2) is zero. For sample measurement, Time Const (tau2) is tau2 from two-tau model.
Sample Coupling	Thermal coupling, in percentage, between sample and platform as determined from two-tau model. 100% indicates perfect thermal contact. 0% indicates no thermal contact. Less than 90% would be considered poor.
Base Samp Temp	Temperature, in kelvin, of sample before heat is applied. Base Samp Temp is determined from fit and is normally identical to set-point temperature.
Avg Samp Temp	Average, in kelvin, of maximum and minimum temperature. Avg Samp Temp is determined from fit. For addenda measurement, Avg Samp Temp is average of maximum and minimum temperature of platform. For sample measurement, Avg Samp Temp is average of maximum and minimum temperature of sample. For two-tau model, average sample temperature may be slightly different from average platform temperature, since heat flow between the two is accounted for by the model.
Temp Rise	Difference, in kelvin, between minimum and maximum temperature in measurement cycle. Represents temperature resolution of measurement.
Fit Quality (Chi Sq)	Determined by mean square deviation of fit from model and expressed as normalized chi square. Since systematic errors are more likely to dominate over random errors, this field is generally more useful as a relative indicator of the fit quality than an absolute indicator. A value that is distinctly larger for a specific measurement, compared to neighboring measurements, may indicate a bad measurement.

(table continues)

Table 4-6. Measurement Fields in Measurement Status Viewer (Continued)

MEASUREMENT FIELD	DEFINITION
Debye Temp	Determined from Sample Heat Cap and user-defined sample information. Debye Temp is computed by inverting Debye relation, which gives phonon specific-heat contribution at a given temperature. The calculation assumes Sample Heat Cap contains only a lattice contribution. If sample was not present or adequate sample information was not defined when data file was opened, Debye Temp is not calculated. Measurement unit is kelvin.

4.5.2 Graph

The Heat Capacity software plots temperature versus time in a graph that appears in the Measurement Status Viewer. The application plots whatever types of temperature data are selected in the four check boxes located above the graph. Any number of check boxes may be selected. The graph in Figure 4-4 plots the fit temperature and measured temperature of the sample platform versus time.

Table 4-7. Temperature Data Viewing Options

OPTION	DEFINITION
Sample Platform Temp (fit)	Temperature of platform, according to model.
Sample Platform Temp (measured)	Measured temperature of platform.
Sample Temp (from fit)	Temperature of sample, according to two-tau model.
Heater Power (measured)	Applied heater power.

- Magnifying the graph.**
To magnify a region of the graph, click on the graph, and then drag the rectangle downward and to the right. As you drag downward, the rectangle indicates the region of the graph that will be enlarged. When you release the mouse button, the plot automatically adjusts to show the temperature measurement points and measurement times for only the region of the graph that is inside the rectangle. To return the graph to its original size, click anywhere on the enlarged graph, and then drag the rectangle upward and to the left. You may also select the **Undo Zoom** button in the Measurement Status Viewer to return the graph to its original size.
- Panning through the graph.**
To pan through the graph, hold down the right mouse button and drag across the graph. You may drag from left to right or from right to left. You select the **Undo Zoom** button in the Measurement Status Viewer to return the graph to its original size.

4.5.3 Message List Box

The message list box at the bottom of the Measurement Status Viewer summarizes, in sequential order, the most recent tasks that the Heat Capacity system has completed or is completing since the Heat Capacity software was started up. The name of the active task or the most recent task is highlighted in the list box. The name of the highlighted task is identical to the name of the task appearing in the bottom status panel in the Heat Capacity control center.

4.6 Raw Data Browser and Slope Analysis

When each new heat capacity value is written to the primary data file, the corresponding temperature response data are also written to the raw, or .raw, data file. As described in section 4.7.4, the .raw data file is created whenever a new data file is opened. It is normally not necessary to view the raw data itself. However, this dialog provides a convenient way to apply the slope analysis techniques described earlier in this chapter (section 4.3.4). This advanced analysis allows observation of high-resolution features, such as first-order transitions, which are not adequately resolved using the standard data fitting technique described in section 4.3.2. Slope analysis also allows rapid characterization of samples using sparse, high-temperature-rise measurements. These capabilities are provided as a post-processing activity which use the .raw file from the standard measurement process as input.

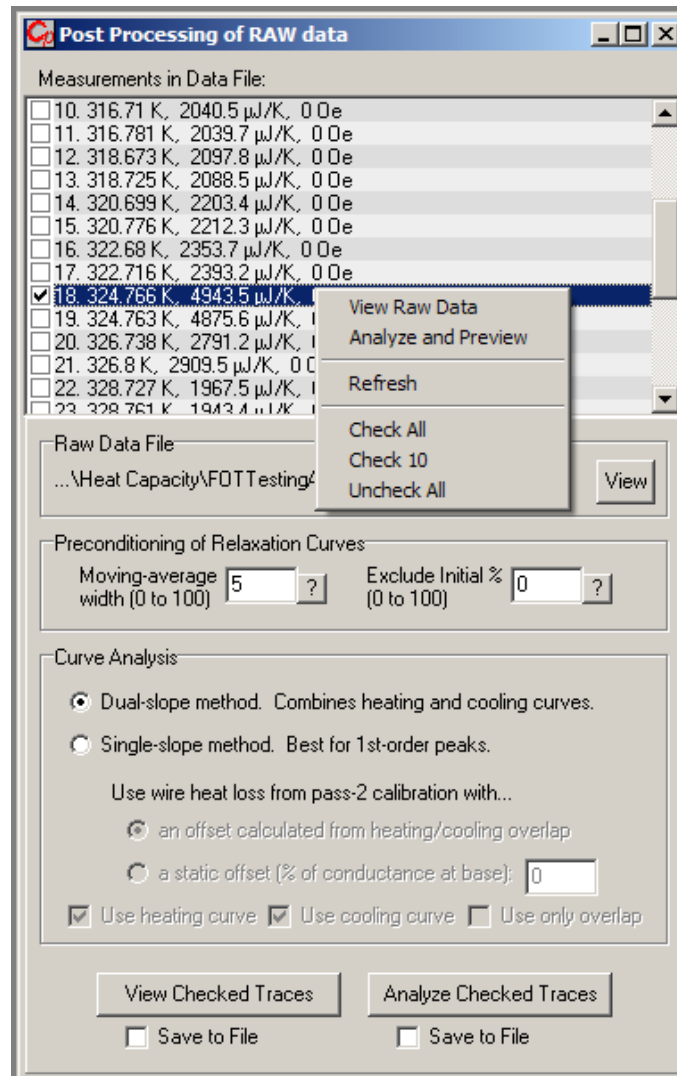


Figure 4-5. Post Processing of Raw Data dialog.

4.6.1 Browsing the Raw Data

To browse the .raw file and to apply the slope analysis, click on **Raw Data File Viewing and Post Processing** button in the **Files** tab (Fig. 4-3) to open the **Post Processing of RAW data** dialog (Fig. 4-5). Note that this button is enabled only if there is a primary data file opened already. To reopen a raw data file, select **Append To...** from the **Files** tab and browse to the primary .dat file containing the data you wish to examine. Once you select the .dat file, the corresponding .raw file will also be opened if it is found in the same directory.

Figure 4-5 shows the **Post Processing of RAW data** dialog. The listing at the top has one line for each relaxation measurement in the raw data file. The average temperature, heat capacity (from the fit analysis), and the magnetic field are indicated on each line. Below the list of measurements is the **View** button to open the .raw file in a MultiVu graph window. This file will contain temperature response data and the corresponding fits for all the measurements in the opened data file. To view a single measurement curve in a MultiVu graph window, right-click on a trace in the list of measurements, and select **View Raw Data**. The raw temperature response curve will be extracted from the .raw data, written to a temporary data file, and displayed in a MultiVu graph. An example of this is shown in Fig. 4-6 from a sample with a possible first-order transition near 327 K. To display the temperature response curves from more than one measurement, check each desired row in the list, then click the **View Checked Traces** button at the bottom of the dialog.

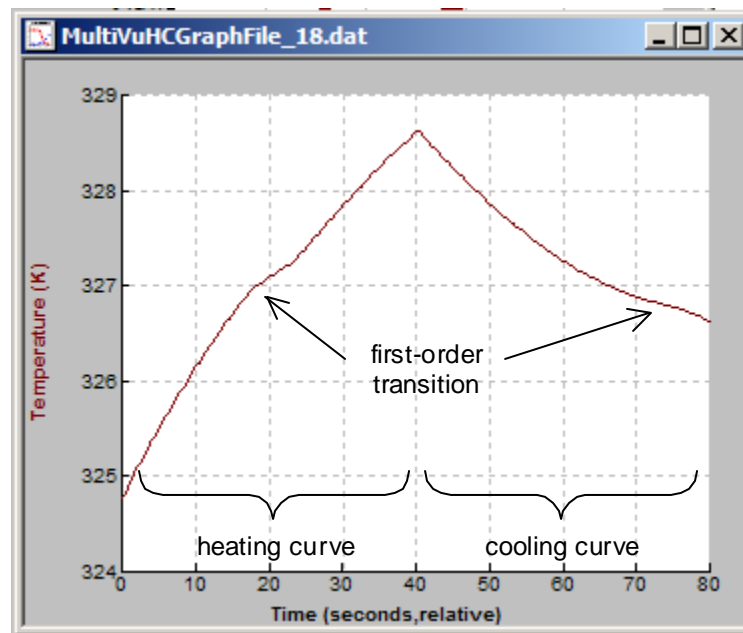


Figure 4-6. Example of raw temperature response data distorted near a first-order transition.

4.6.2 Slope Analysis

The **Curve Analysis** section of the **Post Processing of RAW data dialog** contains parameters for the slope analysis of both the heating and cooling curves of each temperature response. The single-slope method (see section 4.3.4.1) and the dual-slope method (see section 4.3.4.2) are selected using radio buttons. To convert a single temperature response curve to a heat capacity curve using a slope analysis technique, set the parameters for the analysis, right-click on a row in the list of measurements and select the **Analyze and Preview** menu item. The temperature response data will be analyzed and

written to a temporary data file and displayed in a MultiVu graph. To analyze temperature response curves from more than one measurement, check each desired row in the list, then click the **Analyze Checked Traces** button at the bottom of the dialog.

4.6.2.1 EXAMPLE: SINGLE-SLOPE ANALYSIS

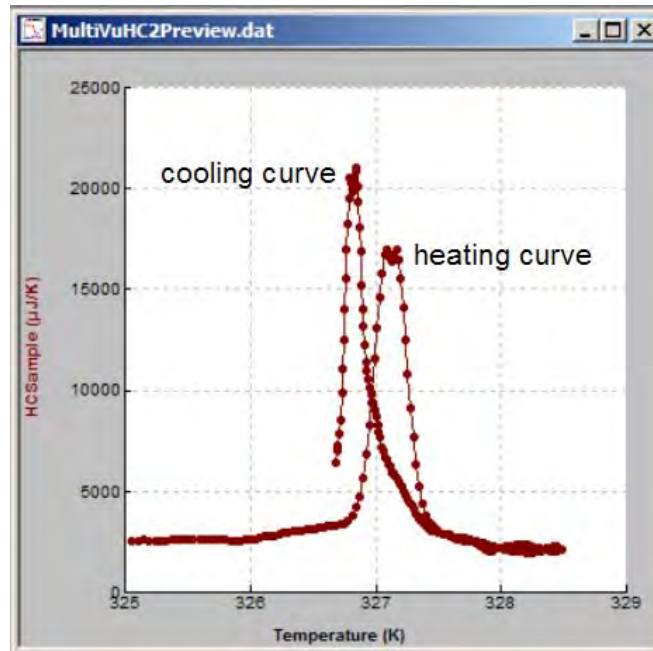


Figure 4-7. Example data from Fig. 4-6 analyzed using the single-slope analysis technique with a Moving Average Width of 10%

Figure 4-7 shows the measurement data from Fig. 4-6 after analyzing the heating and cooling curves with the single-slope method and a **Moving Average Width** (MAW) of 10 percent. The detailed meaning of the MAW parameter is described in more detail in section 4.3.4. Because it is a moving average, neighboring data points are not statistically independent, unless MAW is set to zero. The best value of MAW to use will depend on your specific measurement (and noise level) and will require some trial and error. This is why this analysis is provided as a post-processing analysis and not a measurement parameter. Larger values of MAW will reduce noise and smear transitions. Smaller values will increase noise and sharpen transitions.

The **Exclude Initial %** setting was set to zero in the above analysis. This setting will include all data points when selecting the slope segments of both the heating and cooling curves. However, if your measurement contains significant two-tau effects (sample coupling less than about 95%), it may be helpful to mask the transients in the relaxation curve immediately following the activation and deactivation of the heater. Specifying a non-zero value for “Exclude Initial %” will accomplish this. The best setting will depend on the details of your specific measurement.

When using the single-slope method, the wire conductance from the pass-2 calibration is used when converting the heating and cooling curves into heat capacity curves. The model used for the single-slope analysis is very limited. This is in order to preserve the distinct shape of both the heating and cooling curves as might be needed when analyzing first-order transitions. The single-slope analysis does not attempt to simultaneously solve the heat loss equations. Instead, the heat loss is obtained from the pass-2 calibration data. However, there can be thermal systematic offsets in the conductance numbers present in the calibration file. Also, non-ideal therm coupling between the sample and the platform, or within the sample itself, can cause the apparent effective (or dynamic) heat loss to be

different than pass-2 conductance values. The dual slope analysis and the standard relaxation curve analysis are not very sensitive to such errors, but the single-slope technique is sensitive. To provide a very basic capability to adjust the value of the thermal conductance used in the single-slope analysis, there are two options available. First, you can have the analysis function estimate a conductance correction for the pass-2 conductance that is chosen to minimize the difference between the heating and cooling heat capacity curves. Note that the distinct shapes of the heating and cooling curves are preserved as only a temperature independent offset is calculated and applied to accomplish this. The other option is to manually enter an offset (as a percentage of pass-2 conductance). In this case, you would need to try different values of this offset until the heating and cooling curves have the desired overlap.

It is important to note that when using the single-slope analysis technique (and indeed the dual-slope technique as well), that the heat capacity values obtained will depend on your choice of analysis parameters. These measurement and analysis techniques are not sophisticated enough to provide accurate heat capacity numbers in all cases, or even to estimate the accuracy of the heat capacity numbers obtained. Significant discretion is left to the user in validating the quality of the results and their meaning. An important way to validate the accuracy of slope-analyzed data is to compare the results to the standard relaxation curve-fitting technique (with up to 2% temperature rise).

4.6.2.2 EXAMPLE: DUAL-SLOPE ANALYSIS

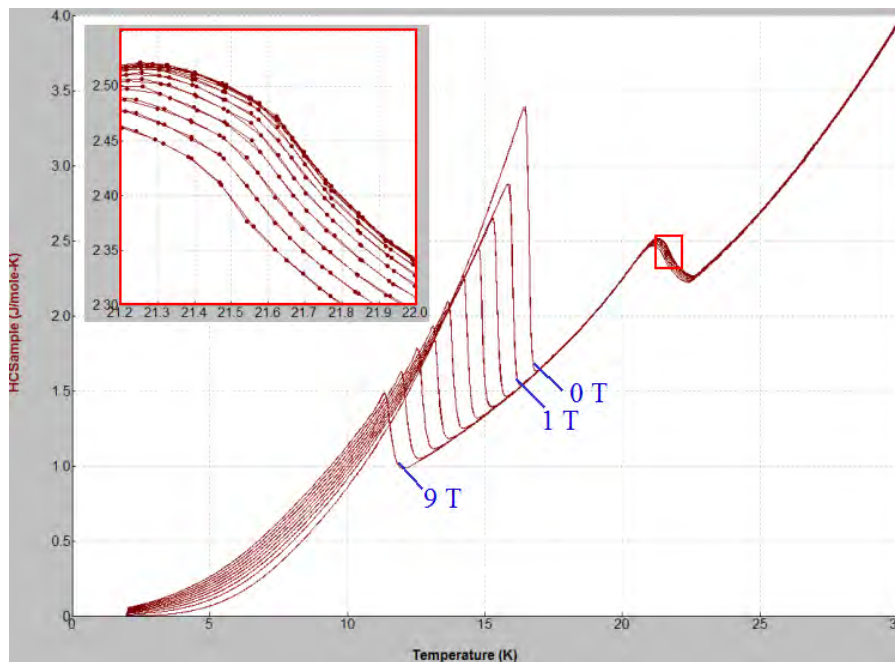


Figure 4-8. Heat Capacity from dual-slope method of a V_3Si superconductor in different magnetic fields. Inset shows the repeatability and resolution that is possible with this method.

Figure 4-8 shows a data set that was analyzed using the dual-slope method. The data from each field was obtained from relaxation measurements at 20 different temperatures. Two relaxation measurements were performed at each temperature using a 30% temperature rise so as to create overlap with neighboring temperature setpoints. Note the excellent overlap of the data as shown in the inset graph.

As is evident from this example, the dual-slope technique is very well suited to high-throughput measurements at high resolutions when sample coupling is good. The accuracy will be degraded somewhat in the case where there is a significant two-tau effect.

4.7 Writing Sequences

Heat Capacity sequence commands can automate all immediate-mode Heat Capacity system operations that are initiated manually from the Heat Capacity control center. For example, by including heat capacity measurement commands and magnet control commands in a single MultiVu sequence file, you can prompt the system to automatically take sample heat capacity measurements at a variety of magnetic fields by just issuing a single PPMS MultiVu **Run** command.

Table 4-8. Heat Capacity Measurement Sequence Commands

SEQUENCE COMMAND	DESCRIPTION	MANUAL REFERENCE
Field Calibrate	Adds magnetic field corrections to calibrations.	Section 5.4
New Addenda	Creates new addenda table.	Section 6.3.6
New Datafile	Switches to new data file.	Section 4.8.1
Puck Calibration Pass 1	Creates new puck calibration file.	Section 5.3.5
Puck Calibration Pass 2	Creates new puck calibration file.	Section 5.3.6
Recalibrate/Verify Table	Recalibrates single thermometer table.	
Sample HC	Measures heat capacity versus temperature.	Section 7.3.9
Switch Addenda	Switches to new addenda table.	Section 6.7.2

For detailed information about creating and editing sequence files and for a discussion about all standard PPMS sequence commands, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual*.

Note: When preparing sequences with Heat Capacity commands such as **New Addenda** or **Sample HC**, it is not necessary to place commands in a **Scan Temperature** loop. The sequence commands themselves implement all the necessary temperature commands and may conflict with any manually defined ones.

4.8 Heat Capacity Data Files

Heat capacity data is normally written to a primary data, or `.dat`, file. Each data file summarizes data from any number of measurements taken for one sample. All heat capacity data saved to a data file may be viewed in MultiVu in real time by selecting the **File>>Open>>DataFile** menu option or the **Open Data File** tool bar button. As each new measurement is completed and the data is written to the file, MultiVu updates its view of the data. New measurement data is always appended to the end of the open data file. The name of the open data file appears in the title bar of the Heat Capacity control center.

For diagnostic purposes and to enable extended post processing data as described above, temperature response data and fit data for each measurement is written to a raw data file. This file has the same name and is located in the same directory as the primary data file, except it ends with a `.raw` extension instead of `.dat`. It is created automatically whenever a new primary data file is opened.

4.8.1 New Datafile Sequence Command

You may switch to a new data file from within an executing sequence by using the **New Datafile** sequence command. In a sequence, you place this command immediately before the addenda or heat capacity command whose generated data you want to save to the file.

Each new file created by the **New Datafile** command uses the current sample header information and current measurement units. If you want to modify the header or file location, you do so before you run the sequence. Once the command is executed, the data file functions like any other data file created with the **Open New File** command.

This sequence command prevents you from overwriting data files. If you assign identical names to more than one data file, the command inserts a numeral after the second and all subsequent occurrences of the file name. For example, the directory of data files may include names like `AddendaLowTemp.dat`, `AddendaLowTemp1.dat`, and `AddendaLowTemp2.dat` after you repeatedly execute the same **New Datafile** command three times.

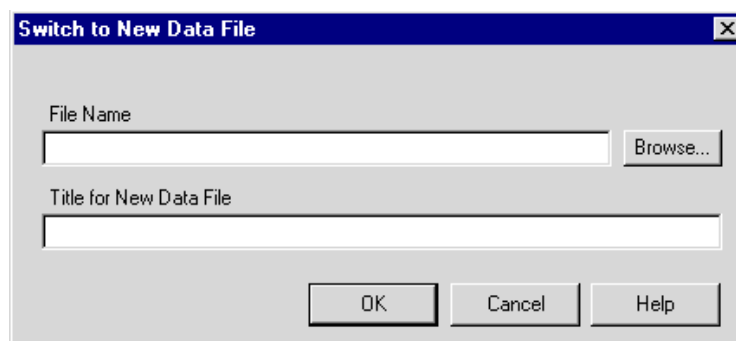


Figure 4-9. **Switch to New Data File** Dialog Box for **New Datafile** Sequence Command

4.8.2 Data File Header

Each primary data file has a file header. The file header contains the sample information that defines one sample. The file header contains four default sample fields—**Mass**, **Masserr**, **Molwght**, and **Atoms**—and it may contain optional, user-defined sample fields (see Section 4.8.2.1). The parameter values in the **Header Information** boxes also create a record of the sample's mass, formula weight, and so on. You may view the header of the active file at any time by selecting the **Sample Info** button in the Heat Capacity control center.

Table 4-9. Default Fields in Header of Data File

FIELD	DESCRIPTION
Mass	Sample mass in mg
Masserr	Uncertainty in sample mass in mg
Molwght	Formula weight in grams per mole
Atoms	Number of atoms per formula unit

The screenshot shows a dialog box titled "Current Data File Header". It contains the following elements:

- Data File Name . . .**: C:\QdPpms\Data\Heat Capacity\HeatCap.dat
- Header Information . . .**: HeatCap.dat
- Buttons**: "Open New File" and "Append to File" are on the right side.
- Table**:

Name:	Description:	Value:
MASS	Sample Mass (mg)	10
MASSERR	Sample Mass Error (mg)	0.1
MOLWGHT	Formula Weight (g/mole)	63
ATOMS	Atoms per Formula Unit	1
- Sample heat capacity units . . .**: µJ/K
- OK** button at the bottom center.

Figure 4-10. Data File Header

You create the file header when you create the data file. Once you create the file, you may not modify its header; the sample information and measurement units permanently reside in the header. The Heat Capacity software uses the parameter values you enter and the measurement units you select in order to express the measurement results as either sample heat capacity or specific heat. The software also uses these parameter values to compute Debye temperature.

4.8.2.1 USER-DEFINED SAMPLE FIELDS

To provide a location for recording additional information in the data file header, the Heat Capacity software allows you to define your own sample information fields. Whenever you create a data file, you are prompted to review, define, and edit the user-defined sample information that is saved to the file header. By default, all user-defined sample information listed under the **Optional Information** heading in the **New Data File Information** dialog box is saved to the file header unless you edit or delete items. Fields you define are not used for any internal computations.

- **Creating a Sample Field.**

When you are creating a data file, you can create a sample field to add to the header of the file. Do the following: (a) select **Configure List** in the **Optional Information** tab of the **New Data File Information** dialog box, (b) select **New Item**, and then (c) use the **Edit User Item** pop-up dialog box to enter the name, default value, and description of the new sample field. Refer to Figure 4-7.

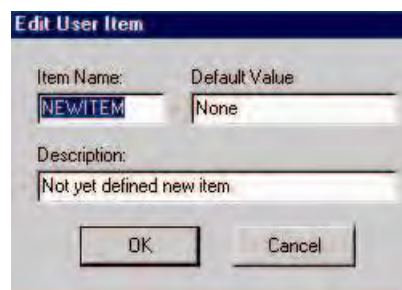


Figure 4-11. **Edit User Item** Dialog Box

- **Editing User-Defined Sample Fields.**

When you are creating a data file, you can edit the user-defined sample fields that will be saved to the header of the file. Do the following: (a) select **Configure List** in the **Optional Information** tab of the **New Data File Information** dialog box, (b) highlight the sample field name, (c) select **Edit**, and then (d) use the **Edit User Item** pop-up dialog box to edit the sample field as necessary.

- **Deleting User-Defined Sample Fields.**

When you are creating a data file, you can delete user-defined sample fields that will be saved to the header of the file. Do the following: (a) select **Configure List** in the **Optional Information** tab of the **New Data File Information** dialog box, (b) highlight the sample field name, and then (c) select **Remove**. Notice that you may not delete the default sample fields.

Note: Different users may have different preferences for user-defined sample fields. To accommodate this, the user information is different for each Windows user. If items in the data file header appear to be missing or different, make sure you are logged on as the correct user.

4.8.3 Format of Data Files

Table 4-10. Fields Visible When Data File Is Open in MultiVu

FIELD	DESCRIPTION
Time Stamp	Time stamp, in seconds. Time Stamp is read from system clock (either Model 6000 clock for PPMS, or PC clock for other systems) immediately following current measurement. When viewed in MultiVu, the field may be converted to minutes or possibly be expressed as relative time, beginning at zero for the first data point in the file.
Comment	Error messages and any changes to the current calibration file and addenda table appear here.
System Status	Integer status code containing temperature, magnet, and chamber status. See <i>Physical Property Measurement System GPIB Commands Manual</i> for PPMS systems.
Puck Temp	Temperature, in kelvin, of puck as determined from the puck thermometer on standard PPMS pucks on standard PPMS systems only.
System Temp	Temperature, in kelvin, of sample interface socket that the calorimeter puck plugs into in the sample chamber or on an installed DR or He3 insert.
Field	Magnetic field, in oersted.
Pressure	Pressure, in torr, measured by sample chamber gauge.
Sample Temp	Identical to value of Avg Samp Temp field in Viewer. See table 4-6.
Temp Rise	Identical to value of Temp Rise field in Viewer. See table 4-6.
Samp HC	Identical to value appearing to left of \pm in Sample Heat Cap field in Viewer. See table 4-6. Measurement units are user selected.
Samp HC Err	Estimated error, in user-selected units, of Samp HC. Samp HC Err is value appearing to right of \pm in Sample Heat Cap field in Viewer. See section 4.3.3.6.
Addenda HC	Identical to value appearing to left of \pm in Addenda Heat Cap field in Viewer. See table 4-6. Measurement units are $\mu\text{J/K}$.
Addenda HC Err	Estimated error, in $\mu\text{J/K}$, of Addenda HC. Addenda HC Err is value appearing to right of \pm in Addenda Heat Cap field in Viewer. See section 4.3.3.6.
Total HC	Identical to value appearing to left of \pm in Total Heat Cap field in Viewer. See table 4-6. Measurement units are $\mu\text{J/K}$.
Total HC Err	Estimated error, in $\mu\text{J/K}$, of Total HC. Total HC Err is value appearing to right of \pm in Total Heat Cap field in Viewer. See section 4.3.3.6.
Fit Deviation	Identical to value of Fit Quality field in Viewer. See table 4-6.
Time Const tau1	Identical to value of Time Const (tau1) field in Viewer. See table 4-6.
Time Const tau2	Identical to value of Time Const (tau2) field in Viewer. See table 4-6.
Sample Coupling	Identical to value of Sample Coupling field in Viewer. See table 4-6.
Debye Temp	Identical to value appearing to left of \pm in Debye Temp field in Viewer. See table 4-6.
Debye Temp Err	Estimated error, in kelvin, of Debye Temp. Debye Temp Err is value appearing to right of \pm in Debye Temp field in Viewer.

(table continues)

Table 4-10. Fields Visible When Data File Is Open in MultiVu (Continued)

FIELD	DESCRIPTION
Cal Correction	Scale factor for platform thermometer resistance. For standard-type pucks, Cal Correction is 1.
Therm Resist	Used only for calibration. Resistance of platform thermometer, in ohms, as measured during first phase of calibration (thermometer calibration). See Section 5.6.1.
Htr Resist	Used only for calibration. Resistance of platform heater, in ohms, as measured during second phase of calibration (thermal conductance calibration). See Section 5.6.2.
Puck Resist	Used only for calibration. Resistance of puck thermometer, in ohms, as measured during first phase of calibration. See Section 5.6.1.
Wire Cond	Thermal conductance, in W/K, of conducting wires as computed from fit.
Meas Time	Duration, in seconds, of measurement. Meas Time is identical to time of furthest right extent of time trace plotted in Viewer in Figure 4-4.
Temp Squared	Square of the Sample Temp field, in K.
Samp HC/Temp	Samp HC field divided by Sample Temp, in user selected units.
Addenda Offset HC	The part of Addenda HC field which is obtained from the offset table. See section 6.7.3.

4.8.4 Format of .raw Files

Table 4-11. Fields visible when the .raw file is open in MultiVu. These fields consist of both raw measured temperature response and calculated fit data.

FIELD	DESCRIPTION
Time	Time axis in seconds, for relaxation measurement.
Comment	The comment field contains parameters for each relaxation measurement. These parameters are contained in a single block before each measurement. See Table 4-12 for an explanation of these parameters.
Therm Res	Resistance, in ohms, of platform thermometer.
Platform Temp	Temperature, in Kelvin, of platform as determined by applying thermometer-resist-calibration table to thermometer resistance value.
Heater Power	Heater power dissipated in platform heater.
Platform Temp Fit	Temperature, in kelvin, of platform from fitting model. Curve matches the Platform Temp field for perfect fit.
Sample Temp Fit	Temperature, in kelvin, of sample computed from fitting model. Field value is identical to Platform Temp Fit only if perfect thermal contact exists between sample and platform.

Table 4-12. Measurement parameters contained in the comment field of the .raw file. These parameters describe the low-level software configuration during the measurement, and the results of the fit analysis.

FIELD	DESCRIPTION
ThResSigmaPerCycle	Sigma, in ohms, per thermometer cycle.
TempSigmaPerCycle	Sigma, in kelvin, per thermometer cycle.
NBinsOn	Number of bins heater is on.
NBinsOff	Number of bins heater is off.
Period	Measurement duration in seconds.
Iterations	Number of times pattern is repeated.
HtrOverLoad	Did heater saturate while measuring?
NCycRec	Number of bridge cycles summed into each bin on each pass.
ThFreqHz	Thermometer bridge measurement frequency.
NSampPerBin	Number of bridge cycles summed into each bin after all passes.
PPMSTemp	Temperature of puck.
EstTau	Pending estimate of time constant before fitting.
ThermOverload	Did thermometer saturate while measuring?
IsAddenda	Is this an addenda measurement?
AddendafromTbl	Addenda heat capacity, in J/K, obtained from active addenda table.
AddendafromTblMinMidMax	Addenda heat capacity at the three temperatures given by TempMinMidMax.
AddendaErrfromTbl	Addenda heat capacity error, in J/K, obtained from active addenda table.
CondfromTbl	Thermal conductance of puck wires, in W/K, obtained from calibration table.
TempMinMidMax	Three temperature values in the neighborhood of the measurement at which AddendaTblMinMidMax and CondfromTblMinMidMax are defined.
CondfromTblMinMidMax	Thermal conductance of puck wires at the three temperatures given by TempMinMidMax.
DisableTau2Fitting	Has dual time constant fitting been purposely disabled for measurement?
HtrCarrOn	Drive current in heater during Heating Curve in amps.
Stable Start Temperature	Temperature of platform before heat pulse.
TimeStamp	See table 4-10.
PPMSStatus	See table 4-10.
PuckTemp	See table 4-10.
SystemTemp	See table 4-10.
Field	See table 4-10.
Pressure	See table 4-10.

SampleTemp	See table 4-10.
TempRise	See table 4-10.
SampHC	See table 4-10.
SampHCErr	See table 4-10.
AddendaHC	See table 4-10.
AddendaHCErr	See table 4-10.
TotalHC	See table 4-10.
TotalHCErr	See table 4-10.
FitDeviation	See table 4-10.
TimeConsttau1	See table 4-10.
TimeConsttau2	See table 4-10.
SampleCoupling	See table 4-10.
DebyeTemp	See table 4-10.
DebyeTempErr	See table 4-10.
CalCorrection	See table 4-10.
ThermCondWire	Same as Wire Cond in table 4-10.
Addenda Offset	Same as Addenda Offset HC in table 4-10.

4.9 Message and Error Log Files

Status messages appearing in the bottom panel of the Heat Capacity control center and in the message list box in the Measurement Status Viewer are written sequentially to the `Messages.txt` file that resides in the `Heat Capacity\LogFiles` directory. The message list box in the Measurement Status Viewer summarizes, in sequential order, the most recent entries in the `Messages.txt` file. In the list box, the active operation or the most recent operation is highlighted. To examine the `Messages.txt` file entries in the Viewer, open the Viewer by selecting the **Meas Status** button in the Heat Capacity control center or by selecting the **View>>Heat Cap Measure Status** menu option. To examine the `Messages.txt` file while a measurement is running, open Microsoft Explorer, and then double-click on `Messages.txt`. The file is reinitialized when you begin the `HeatCap.exe`.

Errors that occur during a measurement and that are displayed in the **Error Message** window are also written to the `Errors.txt` file that resides in the `Heat Capacity\LogFiles` directory. To open the error log file, select the **Error Log** button in the Heat Capacity control center or select the **View>>Heat Cap Error Log** menu option. The `Errors.txt` file is reinitialized when the `HeatCap.exe` starts. Errors are also written to the comment column in the current data file.

Creating and Using Calibration Files

5.1 Introduction

This chapter contains the following information:

- Section 5.2 presents an overview of puck calibration and of calibration files.
- Section 5.3 explains how to calibrate a puck.
- Section 5.4 explains how to add magnetic field correction tables to the calibration.
- Section 5.5 describes the calibration tables.
- Section 5.6 describes the calibration process.
- Section 5.7 discusses calibration file management.

5.2 Overview of Puck Calibration

You must calibrate a puck before you may use it to measure heat capacity. To calibrate a puck, you must run the calibration wizard, which walks you through the process of inserting the calorimeter, identifying the type and serial number, testing the calorimeter, and finally performing the calibration itself. By identifying the type of calorimeter (for example, Helium-3 or standard), you specify the temperature range, thermometer excitation, and other parameters. During the calibration, the Heat Capacity software measures, by using the temperature parameters associated with the puck type, the conductivity of the puck wires and the temperature-dependent resistance of the platform heater, platform thermometer, and puck thermometer. The software stores the serial number, temperature parameters, and measured values in a new calibration, or `.cal`, file. The temperature range defined by the puck type determines the length of time the calibration runs. Although the procedure is automated for non-VersaLab systems, it is necessary to open the chamber between the first and second pass to install the charcoal holder (or to remove the calibration fixture in the case of the Helium-3 insert). The wizard clearly guides you through each of these steps.

Note: If Quantum Design calibrated one or more of the calorimeter pucks supplied with your system, performing the calibration procedure is unnecessary. However, before you use a calibrated puck to measure heat capacity, you must select, as part of the sample installation procedure, the puck's `.cal` file. Refer to Section 7.3 for more information.

At temperatures below about 20 K, the platform thermometer can exhibit a magneto-resistance effect that causes the resistance of the thermometer to change with magnetic field in addition to the normal temperature dependence. The resulting temperature error can also be corrected by using an additional

field-calibration procedure for adding magnetic field correction tables to an existing calibration file. See Section 5.4.

5.2.1 Puck Type

The selected puck type (for example, “Standard”) specifies the parameters that the Heat Capacity software uses to measure the temperature-dependent resistance of the platform heater, platform thermometer, and puck thermometer. These parameter values, which describe the basic physical characteristics of the puck, are stored in a `.pkd` file—for example, `standard.pkd`. Because these parameters are stored in a basic `.pkd` file, design revisions of the puck by Quantum Design can be accommodated by simply distributing a new `.pkd` file.

5.2.2 Calibration Files

The Heat Capacity software saves a puck’s complete calibration data in a single calibration file that resides in a subdirectory under the `Heat Capacity\TempCal` directory. The software assigns the `.cal` extension to the file name. Whenever you calibrate a puck, the software creates a new `.cal` file to store the calibration data. Each `.cal` file contains calibration data for one puck.

A `.cal` file contains the puck’s serial number, the temperature parameters used during the calibration, the measured thermal conductivity and resistance values, the file’s creation date, and the addenda data generated when addenda measurements are taken for the puck (see Chapter 6). The **Calorimeter Files** dialog box (Figure 5-1) conveniently summarizes the `.cal` data. Within the dialog box, each row summarizes the data saved in one `.cal` file. More detailed information about a calibration is displayed in several tables. Section 5.5 discusses these tables.

Before you use a calibrated puck, you select its `.cal` file so that the software matches the correct calibration parameters with the puck. You may easily match a puck with the `.cal` file containing the puck’s calibration data. The software inserts the puck’s serial number immediately before the `.cal` file name extension and displays the serial number in the **Ser#** column of the **Calorimeter Files** dialog box. By default, the software initializes with the last active `.cal` file. Section 5.7.1 discusses how you select a `.cal` file.

The `.cal` files for each different temperature control option (Standard, He3, DR, and VersaLab) are grouped into a different subdirectory under the `Heat Capacity\TempCal` directory. For example, all the `.cal` files for pucks used with the Helium-3 insert are located in the `Heat Capacity\TempCal\He3` subdirectory.

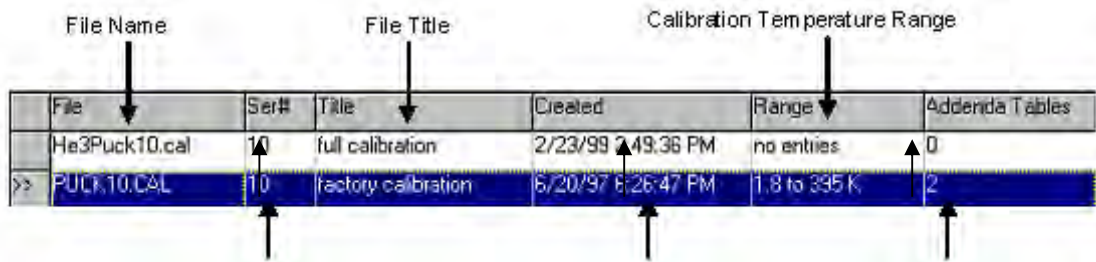
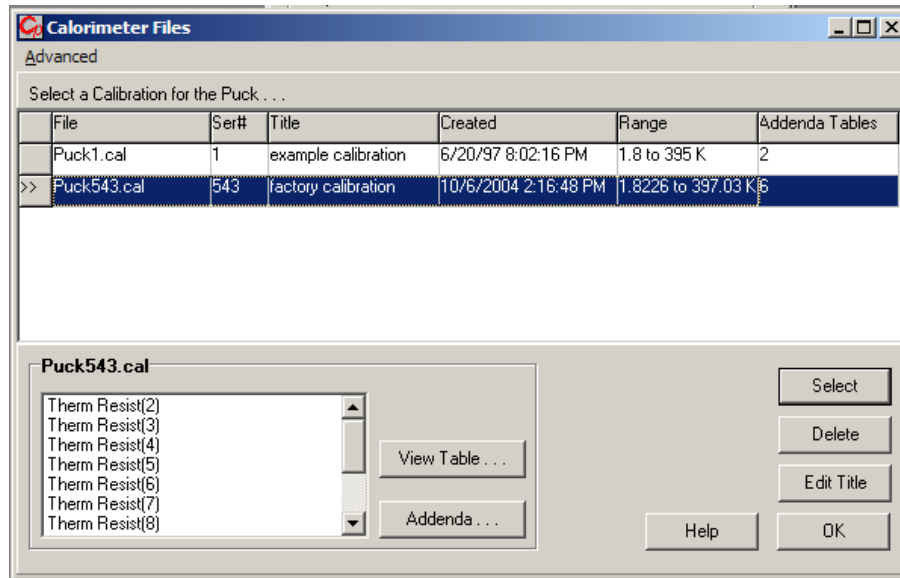


Figure 5-1. **Calorimeter Files** Dialog Box and explanation. To open this dialog, click the “Switch to New...” button on the **Files** tab.

5.2.3 Active Calibration File

The top, or **Calibration**, status panel in the Heat Capacity control center identifies the active .cal file. When you run another calibration or select a different calibration, the name of the .cal file you have selected appears in the status panel.

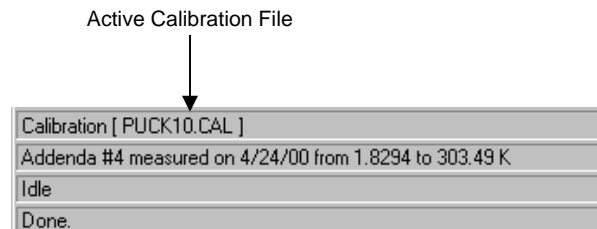


Figure 5-2. Active Calibration File Identified in **Calibration** Status Panel in Control Center

The **Calibration** status panel is also a command button. Double-clicking on the panel opens the **Calorimeter Files** dialog box (Figure 5-1), which contains all .cal files.

5.2.4 Calibration Tables

Each `.cal` file contains a number of tables in addition to other detailed information produced by the calibration procedure. During heat capacity measurements, the Heat Capacity software uses the calibration data in these tables to convert thermometer resistance into temperature values. The software also uses the tables to determine the appropriate amount of heater power and the duration of the measurements. Section 5.5 discusses these calibration tables.

5.2.5 Addenda as Part of Calibration File

The addenda, because it measures the heat capacity of the sample platform that is on the puck, is an extension of puck calibration. The Heat Capacity software stores all addenda data generated for a puck in the puck's `.cal` file. Within the file, the software displays detailed addenda data in separate addenda tables. The **Addenda Tables** column in the **Calorimeter Files** dialog box (Figure 5-1) indicates the number of addenda tables that have been created for the puck. The software uses the active addenda table to calculate the heat capacity of the sample by subtracting the addenda from the total heat capacity.

You measure the addenda any time after you calibrate the puck. Chapter 6 discusses addenda measurements and addenda tables in detail.

5.2.6 Use of Data Files

Calibration data is never lost because the Heat Capacity software saves it to a `.cal` file. However, if you want to use another application, such as MultiVu, to plot the calibration data as it is acquired, you must first open a data file. When you create a data file to save calibration data, you do not define sample information in the data file header. The Heat Capacity software ignores sample information while it calibrates the puck.

5.2.7 Magnetic Field Correction

Note: In order for this calibration procedure to produce the desired field corrections, the system reference thermometer must be corrected for field. There is no benefit to running this procedure if the reference thermometer is not field calibrated. If you are using the Helium-3 or Dilution Refrigerator insert, the insert thermometer, which has been field calibrated by Quantum Design, is the system reference thermometer. Otherwise the sample chamber thermometer is the system reference thermometer. The sample-chamber thermometer is field corrected on all PPMS and DynaCool systems. This field correction is not present, nor is it needed on VersaLab systems. If you are unsure if the sample-chamber thermometer on your system has been field corrected, contact a Quantum Design representative.

Without field correction, the temperature error reported by the Cernox thermometer used on the heat capacity platform ranges from about 1% at 4 K and 90,000 Oe, to more than 30% at 0.5 K.

To calibrate the puck for use in magnetic field, you must first start with a puck that has been calibrated in zero magnetic field. The calibration data at the various magnetic fields is appended to the zero-field data in the `.cal` file. Like the zero-field calibration, the heat capacity magneto-resistance correction depends on the accuracy of the reference thermometer. When performing heat capacity measurements on the Helium-3 system, the reference thermometer corresponds to the Helium-3 system thermometer.

Hence, the Helium-3 thermometer must be fully field corrected for this procedure to work. Similarly, to perform this calibration without the Helium-3 system, the standard block thermometer must be field corrected.

Each time you execute the **Field Calibrate** sequence command, additional field calibration data is saved to the current `.cal` file. Once the thermometer is calibrated at a specific field, repeating it at that field again is unnecessary. The calibration data is permanently stored in the `.cal` file. Additional calibration tables may be added at any time and in any order. Keep in mind, however, that the old calibration data remains as part of the `.cal` even when new calibration data is added at the same field. This may result in unexpected behavior if calibrated more than once at the same field. You may delete unwanted field calibration data from the Advanced menu on the Calorimeter Files dialog (see Fig. 5-1).

Simple linear interpolation is used if a heat capacity measurement is requested at a field that lies between calibration fields. For example, if you execute the **Field Calibrate** sequence command at both 80,000 and 90,000 Oe and subsequently perform a heat capacity measurement at 85,000 Oe, a linear interpolation of the thermometer corrections at the two neighboring fields would be used to synthesize an approximate correction at 85,000 Oe. However, for the best accuracy, it is recommended that measurements be performed at fields corresponding to the calibration values.

If you will be performing HC measurements at arbitrary magnetic fields and you want the best results, you might be tempted to use the **Field Calibrate** command at a very large number of densely spaced fields. However, this is generally not needed and could cause interpolation stability problems if the fields are very close together (within a few percent of each other). The best calibration will approximate the spacing of the underlying magneto-resistance calibration for the base system itself (example: PPMS sample chamber). For the base calibrations, the spacing is typically uniform in the square-root of field with about twice as many field values as the max field in tesla. For example, a typical sample chamber thermometer for a 9 tesla system will have corrections at 290, 1170, 2620, 4670, 7290, 10500, 14300, 18700, 23600, 29200, 35300, 42000, 57000, 65600, 74700, 84300, 90000 Oe. If you create a sequence with the Scan Field sequence command, you would select a spacing code of “H^{1/2}” and perhaps 18 steps starting from 300 Oe and going to 90,000 Oe to obtain a similar spacing. (Don’t use 0 as the starting point, since there is already a calibration at zero field!). Of course, if you will always be performing your HC measurements at a specific set of fields (say, 10000, 20000, 50000, 70000, 90000 Oe), then you might want to just calibrate at these fields.

5.2.8 Compatibility of Electronics: DSP Board and Model 6500 vs. Model CM-E HC Module.

There are two different types of Heat Capacity Controller Electronics. Older PPMS systems use a DSP card that resides in the Model 6500 Option Controller. Newer systems use a Model CM-E Heat Capacity CAN Module. A `.cal` file created with one type of controller CANNOT be used with the other type of controller. If you intend to use pucks that were calibrated with the other DSP controller, you will need to recalibrate them for use with the CM-E HC Module.

5.3 Calibrating a Puck

Note: The following procedures explain how to calibrate a “standard” calorimeter puck. If you are calibrating a calorimeter puck on a Helium-3 or Dilution Refrigerator insert, refer to Chapter 8 for specific instructions.

5.3.1 Prepare the Puck

1. Locate the puck’s serial number, which is written on the green fiberglass connector at the base of the puck (see Figure 3-2). Before you run the calibration procedure, you specify the serial number so that the Heat Capacity software includes the serial number in the .cal file.
2. Remove the sample if a sample is mounted on the sample platform. Refer to Section 7.3.11.
3. Slide the thermal radiation shield over the top of the puck, and then twist the shield to verify that it is securely in position. See warning in Section 11.3.6.
4. Verify that the puck will be in good thermal contact with the bottom of the PPMS sample chamber. Do the following:
 - (a) Make certain the puck fingers are adjusted properly by using the puck adjustment tool (see Section 10.2).
 - (b) Apply a small amount of Apiezon H Grease to the chuck fingers (Figure 3.1) if the fingers are dry. Apply only enough grease to make the fingers slightly sticky. H Grease improves thermal contact between the puck and the socket that the puck is inserted into at the bottom of the sample chamber.

5.3.2 Insert the Puck and Remove the Charcoal Holder

1. Select the **Installation Wizards** tab in the Heat Capacity control center.
2. Select **Prepare New Puck Calibration**. The **Installing Puck for Calibration** dialog box opens.
3. Select **Open Chamber** to vent the sample chamber and warm it to room temperature.
4. Insert the puck into the sample chamber when the on-screen instructions prompt you to do so. The *Physical Property Measurement System: Hardware Manual* discusses puck insertion in detail.
5. Install the baffle assembly but first remove the charcoal holder from the bottom of the assembly. (Removing the charcoal holder is not necessary on a VersaLab system.) Refer to Figure 3-8A.

CAUTION!

Do not touch the charcoal in the charcoal holder. Touching the charcoal reduces its ability to absorb helium at low temperatures. Touch only the gold plate that surrounds the perimeter of the holder.

6. Insert the baffle assembly into the sample chamber.
7. Select **Purge** to purge and seal the sample chamber.
8. Select **Next**. Then define the puck information by (a) entering the puck's serial number and (b) using the drop-down list to select the puck type. If there is more than one choice for puck type, select the one that best describes your system. For example, select **Standard** for standard PPMS or **VLPuck** for VersaLab.
9. Select **Next** to open the **Puck Test Results** panel and initiate the puck test.

5.3.3 Test the Puck

The **Puck Test Results** panel (Figure 5-3) displays the results of a functional test of the electrical connections and the resistance of the puck. The Heat Capacity software measures the resistance, at the current temperature, of the platform heater, platform thermometer, and puck thermometer. The test takes a few seconds and begins as soon as the **Puck Test Results** panel opens. When the measurement is complete, another message, indicating the success or failure of the measurement, appears at the bottom of the panel, and the **Measured** and **Expected** temperatures and resistance values appear in the appropriate columns.

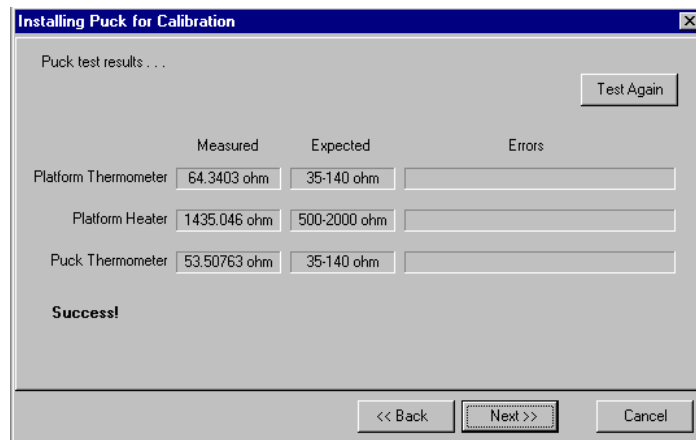


Figure 5-3. **Puck-test results** panel in puck installation wizard. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

A failure might indicate a broken puck wire or a loose or unplugged cable or that the temperature of the puck is changing too rapidly. If a puck wire is broken, you must replace the puck frame. Refer to Section 10.4.1.

- If you want to retest the puck, select **Test Again** in the **Puck Test Results** panel.
- Select **Next** when you are ready to continue. The **Data File Name** panel opens.

5.3.4 Open a Data File

1. Select **Open New File** in the **Data File Name** panel.

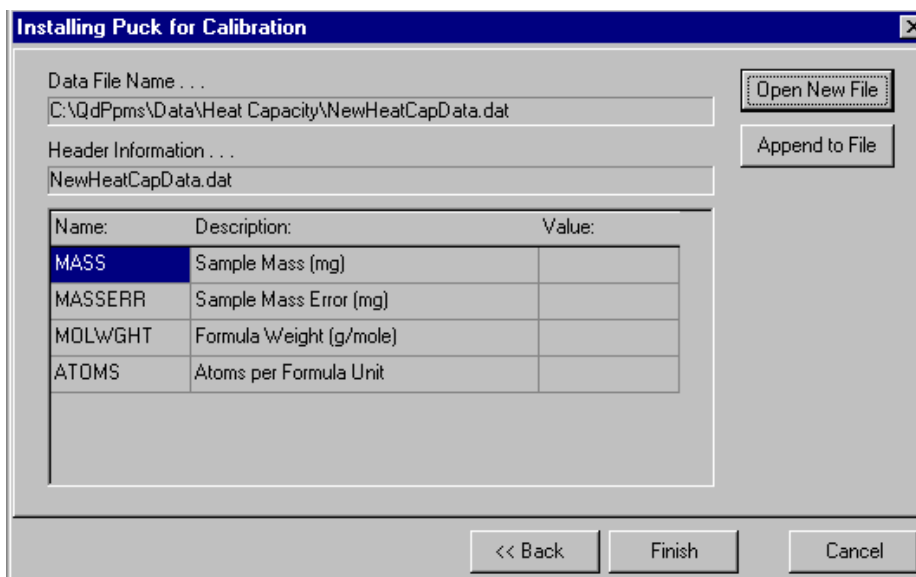


Figure 5-4. **Data File Name** panel in puck installation wizard. The name and location of the active data file are identified at the top of the panel. The information in the header of the active file is displayed below the **Header Information** box. If no data file is active, the **Data File Name** and **Header Information** boxes are blank.

2. Enter the name of the file, and then select **Save**. If you have entered the name of an existing file, a pop-up message asks whether you want to replace the existing file. Select **No**, and then enter another file name.

Once you save the name of the new data file, the **New Data File Information** dialog box opens. The **Optional Information** displayed in this dialog box is the user-defined information that will be saved to the data file header.

3. Enter a different title for the graph view of the file, if necessary. By default, the file name is also the title of the graph view.
4. Review the user-defined **Optional Information** that will be saved to the file header. Select **Configure List** to add, edit, or delete optional items. See Section 4.7.2.1 for more information.

When you create a new data file to store calibration data, you are not prompted to define any sample information that will be saved to the data file header. The application ignores sample information when it runs the calibration.

5. Select **OK** to close the **New Data File Information** dialog box. The **Data File Name** panel appears again. The **Header Information** now displays the header of the data file you have just opened.
6. Select **Finish** to close the **Data File Name** panel and open the **Puck Calibration (Pass 1)** dialog.

5.3.5 Run Pass 1 of the Calibration

Selecting **Finish** in the **Data File Name** panel opens the **Puck Calibration (Pass 1)** dialog box. Verify that the puck serial number and puck type displayed in the **Puck Calibration (Pass 1)** dialog box are correct, and then select **OK** to begin the calibration.

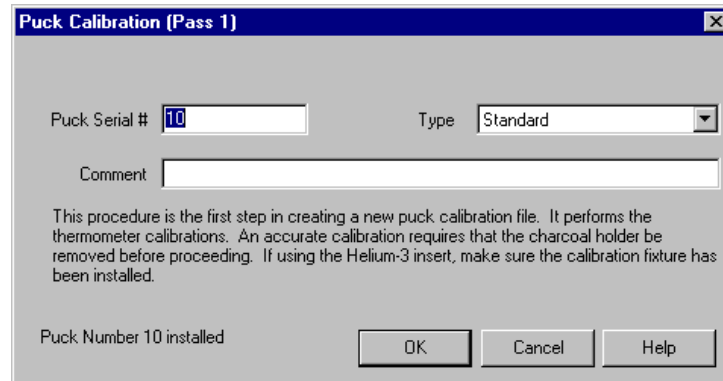


Figure 5-5. Puck Calibration (Pass 1) Dialog Box

When the calibration begins, the Measurement Status Viewer opens to indicate the progress of the calibration. The length of time the first pass runs varies from system to system, but will generally take about 12 hours.

This part of the calibration proceeds from the highest temperature down to the lowest temperature. Section 5.6.1 describes the calibration process in more detail. Once this part of the calibration is complete, the system warms up and leads you through the second pass of the calibration. If you are calibrating a VersaLab puck, the system will continue to pass 2 automatically.

5.3.5.1 PAUSING OR ABORTING A CALIBRATION

You may pause and resume a calibration or abort a calibration at any time.

- To pause a calibration, select **Pause** in the Measurement Status Viewer.
- To abort a calibration, select **Abort** in the Measurement Status Viewer. You may abort a paused calibration.

The **Resume** button in the Viewer is enabled when the calibration is paused.

5.3.6 Run Pass 2 of the Calibration

Once the first pass of the calibration is complete, the system warms up and (for non-VersaLab systems) prompts you to open the chamber and place the charcoal holder back on the baffle assembly. After the chamber is closed again, you purge the chamber as directed, and then you select **Next**. Another puck test is performed. Once it is successful, you select **Next** to proceed. You may select a new data file at this point, or if you want to continue using the same data file, select **Finish**. After selecting **Finish**, you are prompted to begin the second pass of the calibration. Review the comments and select **OK** to begin.

The second part of the calibration begins and the Measurement Status Viewer opens again. This portion of the calibration also starts at the highest temperature and goes to the lowest. It measures the conductance of the wires and heater resistance, and it computes the heat capacity of the bare platform (addenda). Section 5.6.2 describes the calibration process in more detail.

When the second pass of the calibration is complete, the system warms up the sample chamber and prompts you to remove the puck. The PuckXX.cal file is then complete and ready for use.

5.4 Adding Magnetic Field Correction Tables to the Calibration

Once you have a complete .cal file for a puck, you can perform a secondary field calibration on the same puck at the specific fields at which you will be performing measurements. To do this, you execute a sequence that sets the appropriate fields and then executes the **Field Calibrate** sequence command at each value. This procedure appends the necessary calibration data to the .cal file for the puck. This portion of the calibration should only be necessary from about 20 K to the lowest temperature. Do not repeat this procedure for the same field for the same .cal file. See sections 5.2.7 and 5.6.3 for more on performing field corrections.

5.4.1 Example: Calibrating the Helium-3 Puck at 90,000 Oe

1. Install the Helium-3 calibration fixture as described in Section 8.5.1. Installing the calibration fixture ensures that the temperature of the sample platform will be as close as possible to the Helium-3 system thermometer.
2. Select the **Installation Wizards** tab in the Heat Capacity control center.
3. Select **Prepare Addenda Measurement** and follow the on-screen instructions to install the Helium-3 probe in the PPMS sample chamber, select the appropriate calibration file, test the puck, and specify an optional output file. It is assumed that the puck has already been calibrated in zero field so that a corresponding .cal file already exists.
4. Set the field for the PPMS to 90,000 Oe and wait until the field is achieved and the magnet is persistent.
5. Select the **Measurement** tab in the Heat Capacity control center.
6. Select **Calibrate Thermometer in Magnetic Field**.
7. Enter 20 K for the high temperature limit and 0.0 for the low temperature limit (see Figure 5-6). This will generate field correction data at this field from 20 K down to the lowest temperature in the calibration file.
8. Press **OK**. The procedure begins and will take several hours to complete.

Once the procedure is finished, you need to manually set the field back to zero, remove the insert, and then remove the calibration fixture according to the procedures in Section 8.5.3.

When the calibration is complete, you may examine the new calibration table as described in Section 5.5.2.

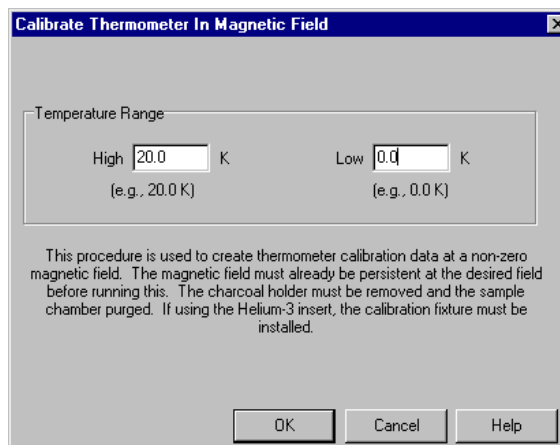


Figure 5-6. Calibrate Thermometer in Magnetic Field Dialog Box

5.5 Description of Calibration Tables

Within the .cal file for a puck, the data describing the temperature dependence of the calorimeter is contained in several calibration tables. Each table contains measured values and the corresponding temperature at which each value was measured. All calibration tables, including addenda tables, for the .cal files can be viewed in the **Calorimeter Files** dialog box. You can open the **Calorimeter Files** dialog box by doing one of the following: (a) double-click on the **Calibration** status panel in the Heat Capacity control center, (b) select **Switch to New** in the **Files** tab in the Heat Capacity control center, or (c) select **Change** in the **Current Calibration File** panel included in the addenda and sample measurement puck installation wizards.

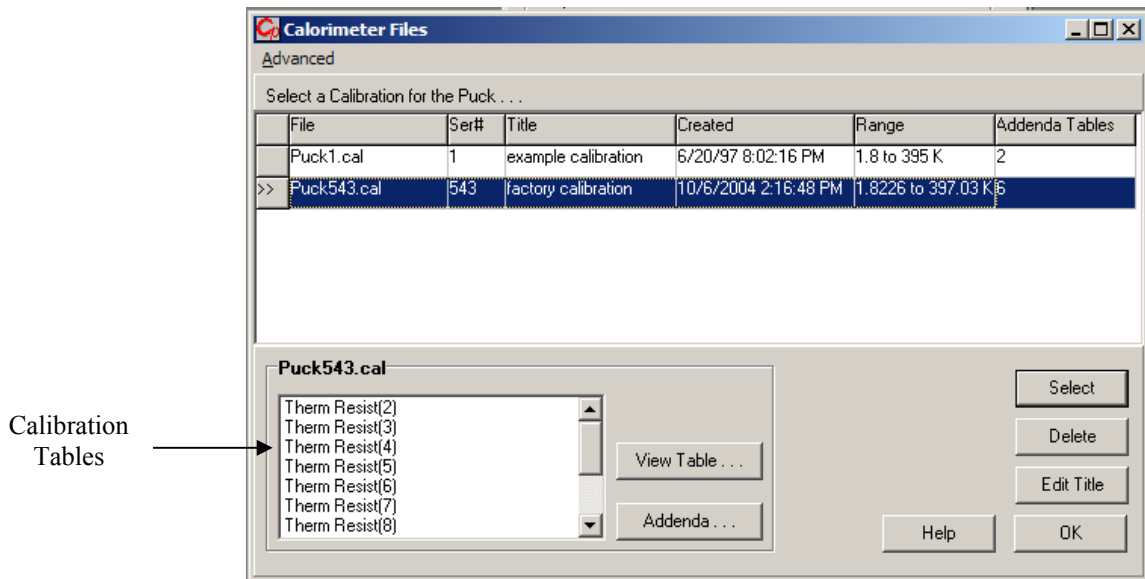


Figure 5-7. List of Calibration Tables in **Calorimeter Files** Dialog Box

5.5.1 Tables for Platform Thermometer Resistance

The Heat Capacity software uses the tables containing the measured values of the platform thermometer resistance for converting the resistance values of the platform thermometer into temperature values. The .cal file may include several of these tables, one for each thermometer current excitation current. A code identifying excitation current follows each **Therm Resist** item in the **Calorimeter Files** dialog box. See Figure 5-7. The smaller the number, the larger the excitation current.

To view a table that displays platform thermometer resistance as a function of temperature, double-click on a **Therm Resist** entry in the **Calorimeter Files** dialog box. In the table, the left-hand column contains the temperature. The right-hand column contains the resistance in ohms.

	Temp	ThRes8
1	1.799868	5267.309
2	2.001415	5274.714
3	2.199378	4556.526
4	2.403486	3990.107
5	2.699893	3385.258

Figure 5-8. Table Displaying Platform Thermometer Resistance

Self-heating effects in the platform thermometer may occur, so the calibration may be slightly different for different measurement currents. To avoid abrupt transitions in the converted temperature, the software uses a different calibration table for each possible excitation current. A transformation is applied to each table after the calibration is complete to ensure that the slope of the calibration curves match at the cross-over from one excitation to the next. The software uses the tables labeled **Therm Curr Code** and the **Therm Gain Code** to determine the appropriate excitation current and readback gains at a given temperature.

5.5.2 Magnetic Field–Corrected Platform Thermometer Resistance

As described above, it is also possible to include thermometer tables that are corrected for magnetic field. Depending on the range of temperatures corrected, many of the **Therm Resist** tables are duplicated for specific fields. For example, if the thermometer was calibrated at 2 tesla, there may be a **Therm Resist (2)** table as well as a **Therm Resist (2, H=20000 Oe)** table. Other fields will be included in a similar way.

5.5.3 Table for Puck Thermometer Resistance

The software uses the table of the puck thermometer resistance values to determine the temperature of the puck while operating at high vacuum. This can help temperature control because the high vacuum can lead to offsets between the PPMS system thermometer and the puck. This table is only present for the standard puck type on PPMS base system. It is not used on DynaCool or VersaLab systems.

To display the values in the table, double-click on **Puck Thermometer Resist** in the **Calorimeter Files** dialog box. The thermometer resistance values are given in ohms.

	Temp	PuckRes
1	1.80003	3340.104
2	1.999763	2864.096
3	2.200169	2506.078
4	2.400087	2230.367
5	2.700013	1918.264
6	3.099962	1621.129

Figure 5-9. Table Displaying Puck Thermometer Resistance

5.5.4 Tables for Platform Heater Resistance and Thermal Conductance

The Heat Capacity software uses these two tables to estimate the amount of heater current that is necessary to produce the requested temperature increase for a measurement.

To display the table containing the platform heater resistance values, double-click on **Heater Resist** in the **Calorimeter Files** dialog box. The heater resistance values are given in ohms. To display the table containing the thermal conductance values, double-click on **Thermal Conductance** in the **Calorimeter Files** dialog box. Thermal conductance is expressed in watts per kelvin.

	Temp	HtrRes
1	1.8	1878.373
2	2.2	1831.836
3	2.7	1786.561
4	3.4	1738.17
5	3.8	1717.723
6	4.7	1679.534

Figure 5-10. Table Displaying Platform Heater Resistance

	Temp	Conductance
1	1.8	1.768562E-6
2	2.2	2.197067E-6
3	2.7	3.33868E-6
4	3.4	4.565116E-6
5	3.8	5.090249E-6
6	4.7	6.845362E-6

Figure 5-11. Table Displaying Thermal Conductance

5.6 Description of Calibration Process

The temperature values at which the system is calibrated, as well as other information about the general characteristics of the puck, are contained in special files that have a `.pkd` extension. When you set up the calibration procedure, you specify the `.pkd` file—by using the **Type** box in the **Puck Calibration (Pass 1)** dialog box—so that the application reads the `.pkd` file while the calibration runs. For example, in Figure 5-5 the `standard.pkd` file is selected. The `.pkd` files are stored in the same directory as the `.cal` files. The following description applies to the standard puck. It is modified slightly for the Helium-3 and Dilution Refrigerator options.

5.6.1 Pass 1

In the first pass of the calibration procedure, the Heat Capacity software first creates a new `.cal` file. The software includes the puck's serial number in the name of the file. For example, if you are calibrating a puck whose serial number is 101, the software creates the `puck101.cal` file. If that file already exists, the software creates the `puck101_1.cal` file, or the software assigns some other unique name that begins with `puck101` to the file. The software never automatically overwrites a `.cal` file. It deletes a file only when you specify that it do so (see Section 5.7.3). Helium-3 `.cal` files begin with the prefix `He3puck` instead of `puck`. Dilution Refrigerator `.cal` files begin with the prefix `DR Puck`, and VersaLab `.cal` files begin with `VLPuck`.

Once the software has created the `.cal` file, it purges and seals the chamber. Purging the chamber cleans the chamber. Sealing the chamber seals it with a small amount of residual helium gas. During the first part of the calibration, this helium gas is necessary for thermal equilibrium among the various thermometers. The charcoal holder is removed for this part of the calibration to ensure that there is always some exchange gas in the chamber, even at the lowest temperatures where the charcoal would normally absorb the helium.

Calibration now begins. Starting from the highest temperature and moving downward, the system stabilizes at each temperature that is within the calibration temperature range defined by the puck type and specified in the `.pkd` file.

When the system stabilizes at a temperature, the software measures and records the resistance of the puck thermometer and platform thermometer. The software uses the PPMS system thermometer as the reference. The results are tables of resistance values versus temperature.

At the end of pass 1 of the calibration, an iterative conditioning algorithm is applied to the individual tables to reduce slope variations and discontinuities at the boundary between thermometer calibration tables at different excitation currents. A backup copy of the unconditioned calibration data is saved in the appropriate `\TempCal` subdirectory with a `.pass1raw` extension.

5.6.2 Pass 2

After the thermometers have been calibrated, you place the charcoal back onto the baffle assembly (standard system only). This ensures that the vacuum conditions in the chamber are the same as the conditions present during a normal heat capacity or addenda measurement; that is, the chamber will be properly evacuated for performing relaxation heat capacity measurements.

The purpose of the second pass of the calibration is to measure approximate values for the thermal conductance of the wire links and also to measure the electrical resistance of the heater on the platform. Although these exact values are not critical for determining the heat capacity of the platform, tables of these values are used during normal heat capacity measurements to help the fitting algorithms converge and to make sure the actual temperature rise values for each measurement are close to the requested values.

When the second pass begins, the system evacuates the sample chamber. Again, starting from the highest temperature and moving downward, the temperature stabilizes at each temperature that is within the calibration temperature range. When the temperature stabilizes, a heat capacity measurement of the platform is performed. The measurement is performed assuming a starting value for the heater resistance and thermal conductance of the wires as contained in the `.pkd` file. The heat capacity measurement itself determines the actual values, which are then saved to the appropriate calibration table in the `.cal` file.

At each temperature, the software writes the partially completed `.cal` file to disk. If the procedure terminates early, a nonfunctional calibration or a partially functional calibration may result. A minimum requirement for functionality of a partial calibration is that at least some portion of the second pass must have completed. Rerun Pass 2 only if this occurs.

5.6.3 Magnetic Field Correction Process

When you run the **Calibrate Thermometer in Magnetic Field** command from the **Measurement** tab in the Heat Capacity control center, the current magnetic field value is recorded in the `.cal` file. Then, based on the temperature range specified, the algorithm compares the temperature range to each of the **Therm Resist** tables (see Section 5.5.1) contained in the current calibration file to determine which tables will be affected. Once the algorithm has determined the list of affected tables, it sorts the temperatures contained in the tables in descending order and then proceeds to measure the resistance of the platform thermometer at each temperature, starting at the highest temperature. Once the **Calibrate Thermometer in Magnetic Field** procedure is complete, the `.cal` file will contain extra tables for the current magnetic field as described in Section 5.5.2. The original tables are not affected. This procedure simply adds information to the `.cal` file.

This procedure does not perform any automatic chamber or magnetic field commands. It is assumed that the charcoal holder has been removed from the contact baffle assembly and the sample chamber has been purged prior to running the procedure. If you are using the Helium-3 or Dilution Refrigerator insert, the calibration fixture must have been installed first. In addition, it is assumed that you have set the magnetic field to the desired field before running the procedure.

5.7 Managing Calibration Files

The Heat Capacity software saves a puck's complete calibration data in a single `.cal` file that resides in a subdirectory under the `Heat Capacity\TempCal` directory. The software creates a new `.cal` file whenever you calibrate a puck. If you use several pucks with the system, you may thus need to manage several `.cal` files. The **Calorimeter Files** dialog box conveniently displays all `.cal` files. Each horizontal row in the dialog box summarizes the data in one `.cal` file. See Figure 5-1.

Section 5.2.2 discusses `.cal` files. Section 5.5 discusses calibration tables.

5.7.1 Selecting a Calibration File

Before you use a calibrated puck, you select its `.cal` file so that the software uses the calibration parameters that have been generated for the puck. To select the correct `.cal` file, you open the **Calorimeter Files** dialog box and then select the row that includes the puck's serial number. When you are preparing to run a heat capacity measurement, the software generates a warning message if the active `.cal` file and the serial number you have entered do not match. The warning message includes instructions that explain how to select the correct `.cal` file.

Complete the following steps to select a `.cal` file:

1. Open the **Calorimeter Files** dialog box (Figure 5-1 or 5-7) by doing one of the following: (a) double-click on the **Calibration** status panel in the Heat Capacity control center, (b) select **Switch to New** in the **Files** tab in the Heat Capacity control center, or (c) select **Change** in the **Current Calibration File** panel included in the addenda and sample measurement puck installation wizards.
2. Locate the `.cal` file that includes the puck's serial number. The serial number appears in the name of the file and in the **Ser#** column. If necessary, adjust the size of the table columns or use the scroll bar to view all information in the columns.
3. Select the correct `.cal` file as follows: (a) click anywhere on the row that summarizes the data saved in the correct `.cal` file, (b) click on **Select**, and then (c) click on **OK** in the pop-up message. The file is selected only when the row is highlighted and the two arrows appear to the left of it.
4. Select **OK** to close the **Calorimeter Files** dialog box.

The calibration data stored in the selected file appears in the **Current Calibration File** panel, and the **Calibration** status panel in the Heat Capacity control center contains the name of the `.cal` file you just selected. Any warning message disappears if the serial numbers match.

5.7.2 Editing the Title of a Calibration File

If you calibrate several pucks and use them with the system, you should assign descriptive titles to the .cal files to help distinguish them. You may edit the title of a .cal file at any time; the file does not need to be active. To edit a title, you select the .cal file in the **Calorimeter Files** dialog box.

Complete the following steps to edit the title of a .cal file:

1. Open the **Calorimeter Files** dialog box (Figure 5-1 or 5-7) by doing one of the following: (a) double-click on the **Calibration** status panel in the Heat Capacity control center, (b) select **Switch to New** in the **Files** tab in the Heat Capacity control center, or (c) select **Change** in the **Current Calibration File** panel included in the addenda and sample measurement puck installation wizards.
2. If you are not editing the title of the active calibration, click once on the row that is part of the .cal file you want to edit. The row is highlighted, but the two arrows do not move to the left-most row column.
3. Select **Edit Title**. Then edit or enter the title as necessary, and select **OK**. The edits appear in the appropriate **Title** column in the **Calorimeter Files** dialog box. If you have edited the title of a nonactive file, the highlight bar returns to the active file.
4. Select **OK** to close the **Calorimeter Files** dialog box.

5.7.3 Deleting a Calibration File

You may delete any .cal file that is not the active calibration. Once you issue the **Delete** command, the .cal file is deleted permanently and may not be retrieved.

Complete the following steps to delete a .cal file:

1. Open the **Calorimeter Files** dialog box (Figure 5-1 or 5-7) by doing one of the following: (a) double-click on the **Calibration** status panel in the Heat Capacity control center, (b) select **Switch to New** in the **Files** tab in the Heat Capacity control center, or (c) select **Change** in the **Current Calibration File** panel included in the addenda and sample measurement puck installation wizards.
2. Click once on any row that is part of a nonactive .cal file. The row is highlighted, but the two arrows do not move to the left-most row column.
3. Select **Delete**, and then select **Yes**. The file name is deleted immediately, and the highlight bar returns to the active file.
4. Select **OK** to close the **Calorimeter Files** dialog box.

5.7.4 Advanced Calibration File Manipulation

By clicking the **Advanced** button in the **Calorimeter Files** dialog there are several commands available for manipulation of .cal file data. These commands must first be enabled by selecting **Enable Advanced Table Operations** from the popup menu.

To view the complete interpolated platform thermometer calibration in a single curve covering the whole temperature range, select the **Advanced >> Export Interpolated Curve from Joined Table** item. This command will ask for a magnetic field at which to extract the calibration curve. The resulting curve will be displayed in a MultiVu graph window. Since the calculated specific heat at a

given temperature is proportional to the slope of this curve, the slope of resistance versus temperature is also displayed. Bumps or other features in this curve may indicate a flawed calibration.

The table conditioning algorithms that are run at the end of pass 1 of the calibration procedure (see section 5.6.1) can be applied manually from the **Advance** menu by selecting **Advanced >>Create Standard Smoothed Version of Active Calibration File**. This procedure will remove kinks and other defects in the calibration data. This is particularly useful when applied to calibration files that were calibrated with an earlier version of the software. It may also be useful if individual tables have been manually edited as described below. Statistical information about the transformation is also generated as part of this operation and is written to a detailed comparison data file for viewing in MultiVu.

Although not recommended, manipulation of individual calibration tables can be performed by opening one of the individual tables as described in Section 5.5. To change a table entry, open the **Calibration Table Edit** dialog (for example, as shown in Fig. 5-8) for the desired table. Then, select the **Edit** tab, change an entry in the text box, then click the **Parse** button. When you are finished, click the **OK** button. This technique can also be used to manipulate addenda tables. See Chapter 6 for more information on addenda tables.

After performing any operations on the calibration file, including editing of the individual tables, you can save or discard the results using the **Advanced >> Save Active Calibration** commands or the **Advanced >> Discard Changes and Reload Calibration** command from the **Calorimeter Files** dialog.

Creating and Using Addenda Tables

6.1 Introduction

This chapter contains the following information:

- Section 6.2 presents an overview of addenda measurement.
- Section 6.3 explains how to measure the addenda.
- Section 6.4 summarizes the addenda parameters.
- Section 6.5 describes the tables for addenda measurement.
- Section 6.6 describes the addenda measurement process.
- Section 6.7 discusses addenda table management.

6.2 Overview of Addenda Measurement

The addenda measurement is the measurement of the heat capacity of the sample platform. The Heat Capacity software calculates the heat capacity of a sample by subtracting the addenda measurement from the total heat capacity measurement. The two measurements—one with and one without a sample on the sample platform—are necessary. In the presence of a magnetic field, it may also be necessary to subtract addenda measurements obtained at each field.

For accurate measurement of the sample heat capacity, you first apply a small amount of grease—just enough to hold the sample—to the sample platform, and then you measure the heat capacity of the grease and the platform: This is the addenda measurement. Next, you mount the sample on the sample platform by pressing the sample onto the grease you have already applied to the platform. You then measure the heat capacity of the sample as described in Chapter 7. Automatic subtraction of the addenda, interpolated at each sample measurement temperature, is performed.

Because each addenda measurement is characteristic of the puck, the Heat Capacity software stores a puck's addenda data in the active calibration, or `.cal`, file. The software creates a new addenda table for each addenda measurement.

6.2.1 Addenda Tables

Addenda tables are stored in the .cal file. The software creates a new addenda table each time you measure the addenda; one addenda table summarizes the addenda data for one measurement. You may take any number of addenda measurements for a puck, and the .cal file may store any number of addenda tables. Only one addenda table is active at a time. By default, the software initializes with the last active addenda table for a given .cal file. You may select any addenda table (see Section 6.7.1) within the active .cal file in order to make that addenda table active.

6.2.2 Active Addenda Table

The **Addenda** status panel in the Heat Capacity control center indicates the active addenda table. The panel displays the date that the addenda was measured and the temperature range of the measurement. When you run another addenda or select a different addenda table, the creation date and temperature range of that addenda table appear in the status panel.

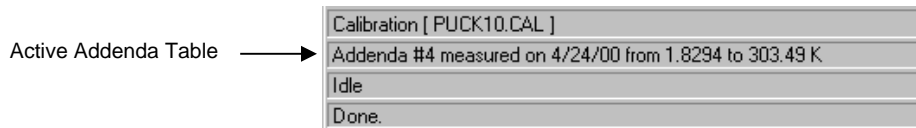


Figure 6-1. Active Addenda Table Identified in **Addenda** Status Panel in Control Center

6.2.3 Use of Data Files

Addenda data is never lost because the Heat Capacity software saves it to the active .cal file. However, if you want to use another application, such as MultiVu, to plot the addenda data as it is acquired, you must first open a data file. When you create a data file to save addenda data, you do not define sample information in the data file header. The Heat Capacity software ignores sample information while it measures the addenda.

6.2.4 Use of Grease

Proper thermal contact between the sample and sample platform requires that grease be used. For accurate measurement of a sample's heat capacity, the grease must be included as part of the addenda. Therefore, you apply grease to the sample platform before you measure the addenda. Use only the amount of grease necessary to attach the sample to the sample platform. The sample's size and geometry determine the correct amount of grease. Generally, one cubic millimeter should be sufficient. If you use too much grease, the poor thermal diffusivity of the grease may yield a poor measurement of the addenda, thus resulting in an inaccurate sample heat capacity measurement. To ensure that the addenda is properly measured, spread the grease out, on the sample platform, in a thin layer over an area that matches the contact area with the sample.

Care is also required during sample mounting to preserve the exact amount of grease present during the addenda measurement. Any discrepancy between the amounts of grease used for the addenda and sample measurements affects the accuracy of the sample heat capacity measurement.

6.2.4.1 RECOMMENDED GREASES

Quantum Design recommends either Apiezon N Grease or Apiezon H Grease¹ for use as an adhesive to attach most samples to the sample platform. Each type of grease has advantages and disadvantages that mainly depend on the range of temperatures over which it will be used.

The consistency of N Grease at room temperature makes it easy to attach samples to the platform and convenient to remove samples from the platform. However, between 260 K and 325 K, N Grease exhibits a slight anomaly in its specific heat near its melting point. This has been documented in the literature (see Bunting, et al, *Cryogenics*, Oct. 1969, pp. 385-386). The heat capacity of N Grease is only reproducible to about 5% in the temperature range 260 K to 325 K. Also, the heat capacity of the grease exhibits peaks in temperature in this range. For best results, use N grease only at temperatures less than 220 K.

Apiezon H Grease has good properties for higher temperature measurements. Since it doesn't melt like N Grease, its specific heat is smooth and reproducible. However, it is more difficult to handle at room temperature, because it does not "wet" the surfaces of the platform or sample very well. Also, at temperatures below 200 K, H Grease has a tendency to spontaneously pop off. So for best results, use H grease for measurements above 200K. For information on the heat capacity of both N and H Grease, refer to "Heat Capacity of Apiezon H Grease from 1 to 50K" by A. J. Bevolo (Bevolo 1974).

If you need to work over the entire temperature range, N Grease is probably the best choice. Although smoothness and accuracy will be worse than with H Grease in the range of the anomaly (260 K to 325 K), overall, the results will be better than H Grease since there will be no loss of grease from it popping off. Improved results can be obtained with N Grease by using the smallest possible amount of grease to hold the sample. Also, the addenda can be sampled densely enough in the range of the anomaly so that the peaks are resolved and can be reasonably subtracted when the sample measurement is performed. An addenda measurement sampled at 5 K intervals between 200 K and 325 K is adequate to eliminate this under-sampling problem.

To summarize:

- Use N Grease at temperatures < 220 K
- Use H Grease at temperatures > 200 K
- For measurements over the entire temperature range, Use N Grease and refer to the above discussion.

6.2.5 Magnetic Field Dependent Addenda

Due to the materials used in the construction of the heat capacity platform, the heat capacity of the addenda depends on magnetic field. The effect is significant only at temperatures below about 10 K, but it is quite large for the original version of the heat capacity platform (see Table 6-1). This effect has been greatly reduced in the current version of the heat capacity platform. You can distinguish between platform versions by their appearance: the original version is opaque white and the current version is translucent (see-through).

¹ Apiezon N Grease and Apiezon H Grease are produced by Apiezon Products Ltd., London, England.

Table 6-1. Comparison between Original and Current Heat Capacity Pucks

Temperature (K)	Original Design		Current Design		
	Addenda at zero field (nJ/K)	Max. addenda change when applying field ² (nJ/K)	Standard puck addenda in zero field (nJ/K)	He-3 puck addenda in zero field (nJ/K)	Max. addenda change when applying field ¹ (nJ/K)
0.4	130	125	N/A	5	1.5
0.6	130	120	N/A	7	2
1	120	100	N/A	15	2
1.5	120	70	N/A	25	2
2.5	180	10	155	90	2
4	470	120	470	310	NM
6	1350	200	1350	1000	NM
10	5400	300	5400	4000	NM

¹ "Maximum addenda change when applying field" is the absolute value of the maximum difference between $C(H)$ and $C(0)$ for the range $0 < H < 16$ T. Note that the maximum difference does not necessarily occur at the maximum magnetic field.

² NM means not measurable within the instrument noise.

To obtain meaningful heat capacity data in magnetic fields when measuring with temperatures below 10 K, you might find it necessary to measure the addenda contribution at the specific fields of interest so that the heat capacity software can perform an accurate addenda subtraction during sample heat capacity measurements. It is especially important to follow this procedure if you are using pucks of the original design, but it usually is not necessary with pucks of the new design. The field dependence of the latter is small enough that you will rarely need to perform field-dependent addenda measurements when you work at temperatures above 1.9 K, and you usually will not need to perform field-dependent addenda measurements when you work at temperatures below 1.9 K.

Rather than measuring the addenda heat capacity in all magnetic fields of interest, you could first measure the addenda only in zero (0) field. If you choose this approach, you should compare the field dependence of your measured sample heat capacity to that of the addenda heat capacity to ensure that the field dependence of the addenda has not significantly affected your data. From your data, calculate the measured change in the sample heat capacity when applying a magnetic field at a few representative temperatures. Compare these values to those in Table 6-1 for your version of the heat capacity puck (original or current version). If the changes in Table 6-1 are comparable to or greater than the change you calculated in the sample heat capacity, then the sample heat capacity data in magnetic field might suffer significant inaccuracy due to the addenda field dependence. Under the latter circumstances, you should perform addenda measurements in field in order to achieve accurate sample heat capacity in a magnetic field.

If you determine that you must perform addenda measurements at each field of interest, be sure to use the appropriate addenda table for each field when you measure sample heat capacity. You can select addenda tables manually or in a sequence, as explained in Sections 6.7.1 and 6.7.2.

6.3 Measuring the Addenda

6.3.1 Apply Grease to the Sample Platform

1. Plug in the small vacuum pump that is supplied with the sample-mounting station. Verify that the sample-mounting station is receiving vacuum.
2. Slide the thermal radiation shield off the puck if the shield covers the puck.
3. Place the puck, with the sample platform facing upward, inside the sample-mounting station's puck holder. Rotate the puck until the index key slips into the notch at the rear of the holder, and then *gently* push the puck into the holder. Verify that the sample platform is properly seated on the platform holder.
4. Pivot the puck interlock arm toward the puck so that it grabs and immobilizes the puck. When the interlock arm is against the puck, vacuum pulls the sample platform downward.

A hissing sound in the puck holder indicates a poor seal between the sample platform and holder. To eliminate the leak, remove the puck from the holder, clean off all debris that is under the puck, and then place the puck back inside the holder.

5. Position the sample-mounting station and the puck below a wide field stereo microscope and a strong light, if desired.

CAUTION!

Caution and a steady hand are required to mount and remove samples. You risk breaking the puck wires unless you work very carefully *and* you feel comfortable using tweezers to manipulate samples under the microscope. If you break a wire, you must replace the entire puck frame and platform along with the wires. Section 10.4.1 explains this procedure.

6. If a sample from a previous measurement is mounted on the sample platform, remove that sample. Use tweezers to gently slide the sample off the platform. Never pull the sample directly up off the platform. Pulling the sample directly upward may pull the platform off its mount and break the wires.
7. Use a toothpick or the broken edge of a cotton-tipped applicator to apply a small amount of grease to the center of the sample platform. *Apply only enough grease to attach the sample to the platform.* Make certain that grease does not extend over the edges of the platform or touch the wires. Section 6.2.4.1 describes recommended greases. (But do not leave behind cotton fibers).
8. Use a cotton-tipped applicator to clean superfluous grease off the platform. Start in the center of the platform, and brush the grease outwards. Make certain that grease does not drip over the edges of the platform or touch the wires.
9. Push the puck interlock arm away from the puck.
10. Pull the puck off the puck holder.
11. Hold the puck so that the sample platform is facing upward. Slide the thermal radiation shield over the top of the puck, and then twist the shield to verify that it is securely in position. (See warning in Section 11.3.6).

Note: Use the puck adjustment tool (see Section 9.2) on the puck after the puck has been inserted in the sample chamber approximately 10 times. By adjusting the tension in the chuck fingers, the puck adjustment tool ensures solid thermal contact between the puck and the heater block located at the bottom of the sample chamber.

12. Use the broken edge of a cotton-tipped applicator or a toothpick to apply a small amount of Apiezon H Grease to each chuck finger (Figure 3.1) if the fingers are dry. Apply only enough H Grease to make the fingers slightly sticky. Make certain that H Grease does not drip between the fingers.
13. Unplug the vacuum pump if you will not immediately reuse the sample-mounting station.

6.3.2 Insert the Puck

1. Locate the puck's serial number, which is written on the green fiberglass connector at the base of the puck (see Figure 3-2). You need to know the serial number to select the .cal file that has been created for the puck.
2. Select the **Installation Wizards** tab in the Heat Capacity control center.
3. Select **Prepare Addenda Measurement**. The **Installing Puck for Addenda Measurement** dialog box opens.
4. Select **Open Chamber** to vent the sample chamber and warm it to room temperature.

5. Insert the puck into the sample chamber when the on-screen instructions prompt you to do so. The *Physical Property Measurement System: Hardware Manual* discusses puck insertion in detail.
6. Install the baffle assembly. Verify that the charcoal holder is attached to the bottom of the assembly. Refer to Figures 3-7 and 3-8B.

CAUTION!

Do *not* touch the charcoal in the charcoal holder. Touching the charcoal reduces its ability to absorb helium at low temperatures. Touch only the gold plate that surrounds the perimeter of the holder.

7. Insert the baffle assembly into the sample chamber.
8. Select **Purge** to purge and seal the sample chamber.
9. Select **Next**. Then enter the puck's serial number.
10. Select **Next** to open the **Current Calibration File** panel.

6.3.3 Select a Calibration File

Review the information identifying the active .cal file in the **Current Calibration File** panel (see Figure 6-2). The panel displays summary information—serial number, creation date, name, title, temperature range, and the number of associated addenda tables—for the active .cal file. The serial number identified in the **Current Calibration File** panel should match the serial number you entered previously. A warning message appears at the bottom of the panel if the two numbers do not match.

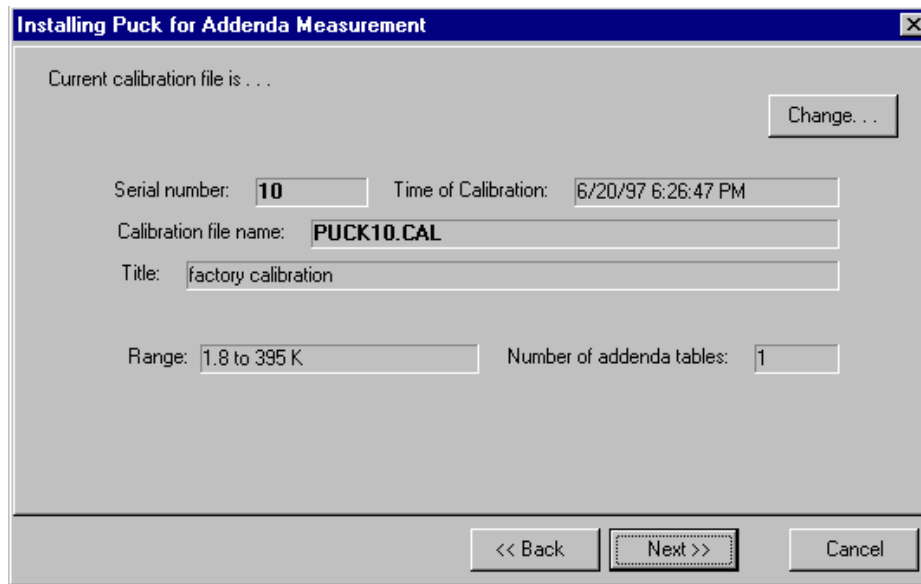


Figure 6-2. **Current Calibration File** Panel in Puck Installation Wizard

- If the serial numbers match, indicating that the correct .cal file is active, select **Next** to open the **Puck Test Results** panel.
- If the serial numbers do *not* match, indicating that the correct .cal file is not selected, you must select the correct .cal file. Do the following:
 1. Select **Change** in the **Current Calibration File** panel. The **Calorimeter Files** dialog box opens (see Figure 5-1 or 5-7).
 2. Locate the .cal file that includes the puck's serial number. The serial number appears in the name of the file and in the **Ser#** column. If necessary, adjust the size of the columns or use the scroll bar to view all information in the columns.
 3. Select the correct .cal file as follows: (a) click anywhere on the row that summarizes the data saved in the correct .cal file, (b) click on **Select**, and (c) click on **OK** in the pop-up message. The file is selected only when the row is highlighted and the two arrows appear to the left of it.
 4. Select **OK** in the **Calorimeter Files** dialog box. The **Current Calibration File** panel now displays all data stored in the .cal file you have activated. Any warning message disappears if the serial numbers match.
 5. Select **Next** to open the **Puck Test Results** panel.

6.3.4 Test the Puck

The **Puck Test Results** panel displays the results of a functional test of the electrical connections and the resistance of the puck. The Heat Capacity software measures the resistance, at the current temperature, of the platform heater, platform thermometer, and puck thermometer. The test takes a few seconds and begins as soon as the **Puck Test Results** panel opens. When the measurement is complete, a message, indicating the success or failure of the measurement, appears at the bottom of the panel, and the **Measured** and **Expected** temperatures and resistance values appear in the appropriate columns. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

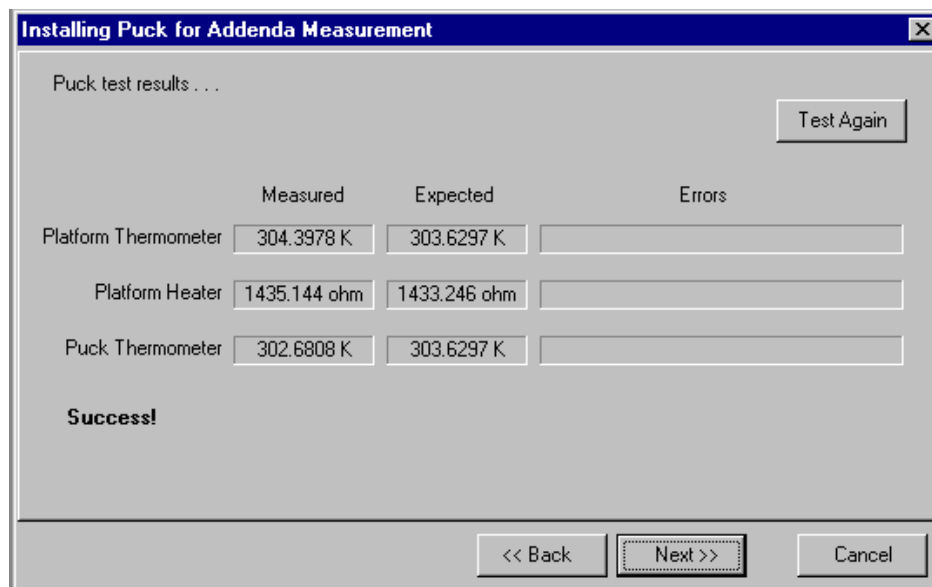


Figure 6-3. **Puck Test Results** panel in puck installation wizard. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

A failure might indicate a broken puck wire or a loose or unplugged cable or that the temperature of the puck is changing too rapidly. If a puck wire is broken, you must replace the puck frame. Refer to Section 9.4.1.

- If you want to retest the puck, select **Test Again** in the **Puck Test Results** panel.
- Select **Next** when you are ready to continue. The **Data File Name** panel opens.

6.3.5 Select a Data File

Note: Opening a data file to save the addenda measurement data is optional. However, if you want to plot the addenda data, you must save it to a data file. You may use the currently selected file, create a new file, or append data to an existing file.

Note: In certain cases, it might be useful to append new measurements to the end of an existing file. When you append to an existing file, the original header information and the measurement units that were originally selected are preserved in the file.

Review the **Header Information** in the **Data File Name** panel. The **Header Information** lists the information stored in the header of the active data file. The **Header Information** is blank if a data file is not open.

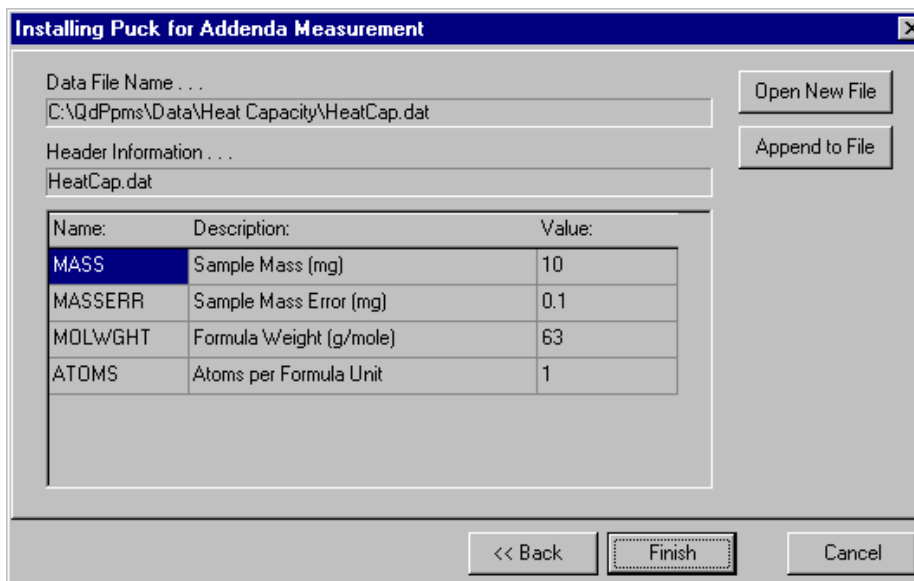


Figure 6-4. **Data File Name** panel in puck installation wizard. The name and location of the active data file are identified at the top of the panel. The information in the header of the active file is displayed below the **Header Information** box. If no data file is active, the **Data File Name** and **Header Information** boxes are blank.

- If you want to use the data file that is currently active, select **Finish**. The **Data File Name** panel closes.
- If you want to append data to a different data file, select **Append to File**, select the file, and then select **Open**. The **Header Information** in the **Data File Name** panel lists the information stored in the header of the file you have just selected. Select **Finish** to close the **Data File Name** panel.
- If you want to create a new data file, do the following:
 1. Select **Open New File** in the **Data File Name** panel.

2. Enter the name of the file, and then select **Save**. If you have entered the name of an existing file, a pop-up message asks whether you want to replace the existing file. Select **No**, and then enter another file name.

Once you save the name of the new data file, the **New Data File Information** dialog box opens. The **Optional Information** displayed in this dialog box is the user-defined information that will be saved to the data file header.
3. Enter a different title for the graph view of the file, if necessary. By default, the file name is also the title of the graph view.
4. Review the user-defined **Optional Information** that will be saved to the file header. Select **Configure List** to add, edit, or delete optional items. See Section 4.7.2.1 for more information.

When you create a new data file to store addenda measurement data, you are not prompted to define any sample information that will be saved to the data file header. The software ignores sample information when it measures the addenda.
5. Select **OK** to close the **New Data File Information** dialog box. The **Data File Name** panel appears again. The **Header Information** now displays the header of the data file you have just opened.
6. Select **Finish** to close the **Data File Name** panel.

6.3.6 Run the Addenda Measurement

1. If you will measure addenda in a magnetic field, you must first set the magnet persistent at the desired field. When the addenda measurement begins, the current field will be recorded for subsequent heat capacity measurements.
2. Select the **Measurement** tab in the Heat Capacity control center.
3. Select **Create New Addenda Table**. The **Create New Addenda Heat Capacity Table** dialog box opens. You use this dialog box to specify the parameters for the addenda measurement. The dialog box displays the values used for the last addenda measurement. The parameters are organized into a **Setup** tab (Figure 6-5) and an **Advanced** tab (Figure 6-6). Section 6.4 explains the parameters in more detail.

Note: Under certain circumstances, residual helium may be absorbed by components of the calorimeter if the calorimeter is left below 6 K for several hours prior to a measurement. If this is the case, it is recommended that the system be thermally cycled to above 6 K prior to a measurement or that you measure with decreasing temperatures starting above 6 K.

4. Select the **Setup** tab. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

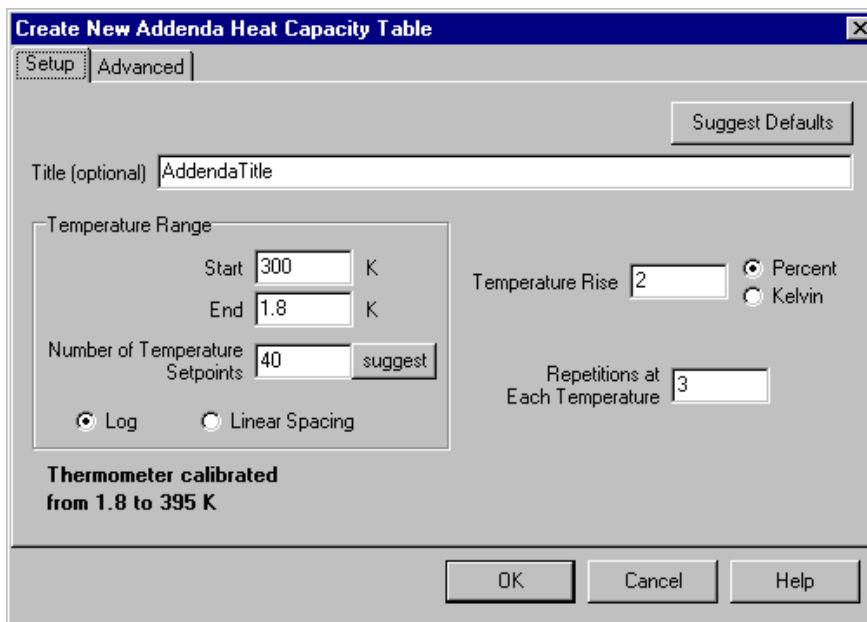


Figure 6-5. Setup Tab in Create New Addenda Heat Capacity Table Dialog Box

5. Enter the title of the addenda table. Make the title as descriptive as possible so that you can match the addenda table with the correct sample.
6. Enter the starting value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range, which are displayed in the lower left corner of the dialog box. The first measurement is taken at the starting temperature value.
7. Enter the ending value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range. The last measurement is taken at the ending temperature value.
8. Enter the number of temperature values, including the starting and ending temperature values, for which you want to take measurements. Pressing the **suggest** button inserts a value large enough to ensure good interpolation, yet small enough to avoid unnecessary over-sampling. See Section 13.3.3 for special considerations when using N grease in the 200K to 300K range.

Note: Selecting exactly matching measurement points for the sample heat capacity measurement and the addenda measurement is unnecessary. During the sample heat capacity measurement, the software uses polynomial interpolation of the active addenda table at the measurement temperatures for determining the appropriate addenda to subtract.

9. Select the type of spacing that is used to separate the temperature values.
10. Enter a value for the temperature rise.
11. Enter the number of times a measurement is repeated at each temperature.
12. Select the **Advanced** tab if you want to review the advanced measurement parameters. Quantum Design recommends that only default advanced measurement parameters be used. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

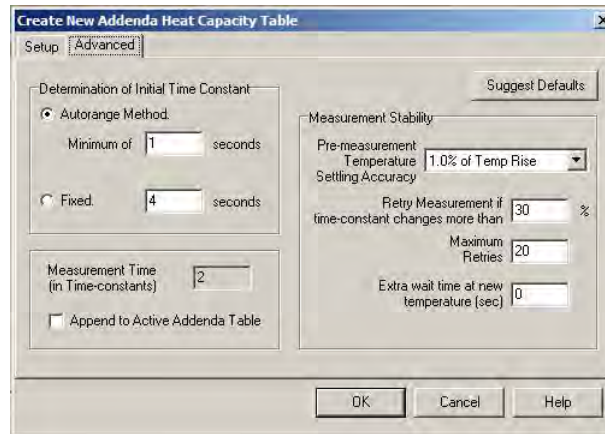


Figure 6-6. **Advanced** Tab in **Create New Addenda Heat Capacity Table** Dialog Box

13. Select the **OK** button at the bottom of the **Create New Addenda Heat Capacity Table** dialog. The addenda measurement begins if the temperature range you defined is within the calibrated temperature range. If the temperature range is outside the calibrated range, a warning message that lists the suggested values pops up. You may override the warning and run the addenda.

As soon as the addenda begins to run, the Measurement Status Viewer opens to show the measurement's progress. The name of each task that is part of the measurement appears, as it is performed, in the message list box at the bottom of the Viewer. When the software performs a new measurement, it presents the data in the measurement-field panels and plots the data as a graph. Section 4.5 discusses the Measurement Status Viewer in detail.

The bottom status panel in the Heat Capacity control center also indicates the name of each task as the task is performed.

Section 6.6 describes the addenda measurement process in more detail.

14. Wait for the measurement to finish. A measurement generally lasts several hours. When the measurement is complete, the **Idle** and **Done** messages appear in the bottom status panels in the Heat Capacity control center.

6.3.6.1 PAUSING OR ABORTING A MEASUREMENT

You may pause and resume an addenda measurement or abort an addenda measurement at any time.

- To pause a measurement, select **Pause** in the Measurement Status Viewer.
- To abort a measurement, select **Abort** in the Measurement Status Viewer. You may abort a paused measurement.

The **Resume** button in the Viewer is enabled when the measurement is paused.

6.3.7 Remove the Puck

Refer to the *Physical Property Measurement System: Hardware Manual* to remove the puck from the sample chamber.

6.4 Summary of Addenda Parameters

Table 6-2. Parameters in **Setup** Tab in **Create New Addenda Heat Capacity Table** Dialog Box

PARAMETER	DEFINITION
Title	Descriptive title for identifying this addenda in Addenda Tables dialog.
Temperature Range Starting Value	Starting value for temperature range. First measurement is taken at this temperature. Addenda temperature range should be within temperature range of thermometer calibration.
Temperature Range Ending Value	Ending value for temperature range. Last measurement is taken at this temperature. Addenda temperature range should be within temperature range of thermometer calibration.
Number of Temperature Values	Number of temperature values, including starting and ending value, at which measurements are taken. Pressing suggest button inserts a value large enough to ensure good interpolation, yet small enough to avoid unnecessary over-sampling.
Spacing for Temperature Values	Logarithmic or linear spacing between temperature values. Logarithmic spacing equally spaces logarithms of temperature values. Linear spacing equally spaces temperature values.
Temperature Rise	<p>Temperature rise produced by heater-on cycle of heat capacity measurement. Large temperature rise reduces scatter. Small temperature rise is necessary if features such as peaks must be resolved. While there should be no such features in the addenda itself, it is a good idea to use the same temperature rise for the addenda and subsequent heat capacity measurement. A typical value for temperature rise is 2%.</p> <p>Temperature rise is either given in kelvin or as a percentage of sample temperature at each measurement, depending on user selection. The application automatically determines the heater current needed to produce this temperature rise.</p>
Number of Repetitions Per Measurement	Number of times a measurement is repeated at each temperature. A value of two or more is recommended since this gives good indication of self-consistency of measurements.

Table 6-3. Parameters in **Advanced** Tab in **Create New Addenda Heat Capacity Table** Dialog Box

PARAMETER	DEFINITION
Determination of First Time Constant: Autorange Method	If this option is selected, initial time constant is determined by applying a constant heater power and determining amount of time it takes for temperature to rise to half the requested delta. The minimum value is used if the resultant time constant is less.
Determination of First Time Constant: Fixed	If this option is selected, initial time constant is the specified value. Use this if you already roughly know (less than a factor of two or so) the time constant from previous measurements at this temperature. The auto ranging step will be skipped, thus obtaining first measurement more quickly.
Relaxation Duration: Measurement Time (In Time Constants)	Amount of time, including heater-on and heater-off sequences, used for a single relaxation measurement. For “standard” puck type, this value is always 2.
Append to Active Addenda Table	When selected, the addenda measurements are appended to current addenda table instead of creating a new table. Only temperatures that extend (above or below) the existing table are allowed. This is useful for continuing an aborted run, or for creating non-uniform addenda spacing. (For example, see section 12.3.3.)
Measurement Stability: Settling Accuracy	Determines how long software waits for temperature stability before taking each measurement. Settling accuracy is given as a percentage of temperature rise. If sample-platform temperature changes between successive readings by less than the settling-accuracy value, temperature is stable enough to proceed with measurement. Small settling accuracies lead to better temperature reproducibility at a specific temperature, though not necessarily better heat capacity values. That is, the temperature scatter will be smaller when this value is smaller but heat capacity values will still fall on correct curve. However, the measurement will take longer with a smaller value. A typical value for settling accuracy is 1%.
Measurement Stability: Retry Percentage	Determines how much the time constant is allowed to change from measurement to measurement. For example, a typical value of 30% would mean that if previous relaxation measurement had a time constant of 1 second, and the current measurement was greater than 1.3 or less than 0.7, the current measurement would be discarded and the measurement repeated with the latest acquired time constant. A smaller value may reduce temperature hysteresis effects with some samples by requiring better self-consistency between the assumed time constant used for the “Measurement Time” and the actual time constant determined from the fit.
Measurement Stability: Maximum Retries	Maximum number of times the above condition will discard a measurement before doubling the user-specified “Measurement Time.” This is an attempt to recover self-consistency. The assumption is that the sample is possibly very poorly attached or that specified Measurement Time is much less than 1 and is too short to discern a good value for the time constant when the data is fit. All subsequent measurements will be performed with the longer time constant.
Measurement Stability: Extra wait time at new temperature (sec)	After the PPMS declares stability at a new temperature, the software will wait for the indicated time before starting the measurement. Subsequent measurements at the same temperature do not wait.

6.5 Description of Addenda Tables

The addenda table for any addenda measurement contains two separate tables—one displaying the heat capacity values of the sample platform, and one displaying error bar values—plus an optional user-defined offset table. When the Heat Capacity software measures the heat capacity of a sample, it determines the errors from the fitting algorithm. The offset table, which is read from a separate file in the TempCal directory, is used in cases where a known heat capacity from, for example, an auxiliary sample holder, should also be included in the addenda. Section 6.7.3 discusses offset tables in more detail.

You view any addenda table by selecting the appropriate command button in the **Addenda Tables** dialog box. You open the **Addenda Tables** dialog box (Figure 6-9) by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.

- To view the table of addenda heat capacity values versus temperature, select **Addenda Entries** in the **Addenda Tables** dialog box.
- To view the error table, select **Addenda Error Entries** in the **Addenda Tables** dialog box.
- To create the optional user-defined offset table, select **Addenda Offset Formula** in the **Addenda Tables** dialog box.

The screenshot shows a dialog box titled "Calibration Table Edit" with a menu bar containing "File". Below the menu bar is a table with two columns: "Temp" and "AddendaHC". The table contains six rows of data. Below the table is a "Calculator" section with two input fields: "Temp" and "AddendaHC", both with "(no pre-function)" below them. Between the fields are two buttons: "==" and "<==". At the bottom are "OK" and "Cancel" buttons.

	Temp	AddendaHC
1	1.829363	0.01001167
2	2.082635	0.0148385
3	2.371341	0.02199852
4	2.701166	0.03260193
5	3.075822	0.04831461
6	3.503663	0.07162027

Figure 6-7. Table Displaying Addenda Entries

The screenshot shows a dialog box titled "Calibration Table Edit" with a menu bar containing "File". Below the menu bar is a table with two columns: "Temp" and "AddendaHCErr". The table contains six rows of data. Below the table is a "Calculator" section with two input fields: "Temp" and "AddendaHCErr", both with "(no pre-function)" below them. Between the fields are two buttons: "==" and "<==". At the bottom are "OK" and "Cancel" buttons.

	Temp	AddendaHCErr
1	1.829363	4.337284E-6
2	2.082636	5.974088E-6
3	2.371341	1.561693E-5
4	2.701166	2.675387E-5
5	3.075822	2.857165E-5
6	3.503663	0.0001165942

Figure 6-8. Table Displaying Addenda Errors

6.6 Description of Addenda Measurement Process

The Heat Capacity software uses similar procedures to acquire data for addenda measurements and sample heat capacity measurements. The main differences between the two types of measurements are how the software converts raw data to a heat capacity value and how the software saves heat capacity data. Section 7.6 describes the sample heat capacity measurement process.

When an addenda measurement begins, the vacuum system first evacuates the sample chamber. Then the chamber temperature stabilizes at the user-defined starting temperature value. Measurements begin when the temperature is stable. The software measures the temperature response of the sample platform to the applied heat, and then fits the acquired time trace to a model that assumes a sample is not present; that is, a simple model (Section 4.3.1.1) is used for the fit. The use of this model is an important difference between the analysis of the data for determining the addenda heat capacity and the sample heat capacity.

Once the software has determined the heat capacity value and the associated error bar, it writes these values to a new addenda table residing in the active `.cal` file. A new addenda table is created each time you run the procedure. You can append data to an existing table if you select the **Append to Active Addenda Table** option on the **Advanced** page as described in Table 6-3. The current magnetic field value is also written to the `.cal` file and appears in the **Addenda Tables** dialog box. The software never automatically overwrites an addenda table and deletes a table only when you specify that it do so. Section 6.7.5 explains how to delete an addenda table.

If a data file is open before the addenda measurement begins, the software writes all fit information and the heat capacity of the addenda to the data file. However, when the software measures the heat capacity of a sample, it uses only the information written to the addenda table to compute the sample heat capacity.

6.7 Managing Addenda Tables

The software saves the addenda table inside the active `.cal` file. The **Addenda Tables** dialog box conveniently displays all addenda tables created for the active `.cal` file. Each horizontal row in the dialog box summarizes the data in one addenda table.

You open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.

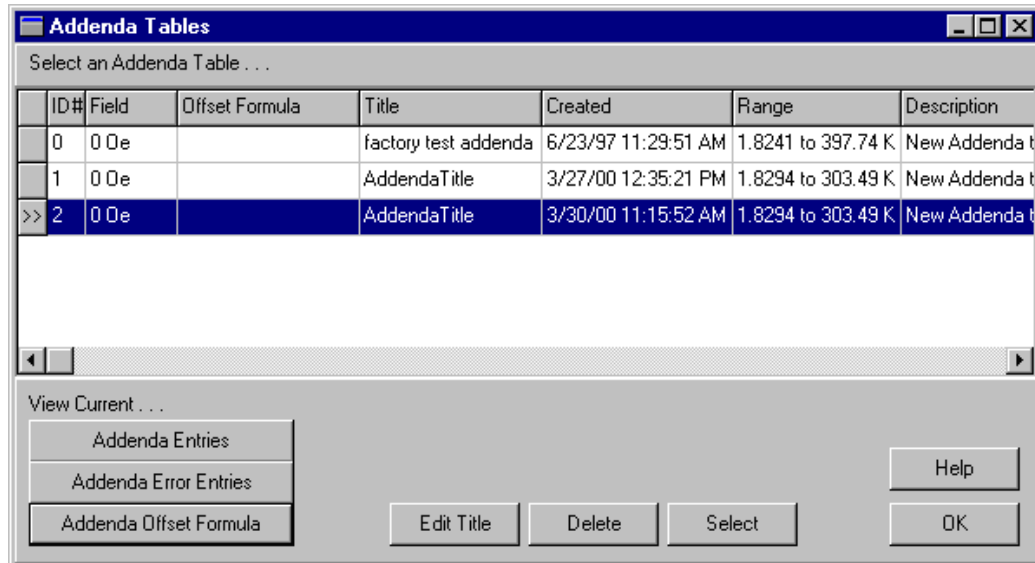


Figure 6-9. **Addenda Tables** Dialog Box

A .cal file may include any number of addenda tables, although only one addenda table is active at a time. The **Addenda Tables** dialog box indicates which addenda table is active. You may select any other addenda table to make it active.

The magnetic field at which the addenda was acquired is included as part of the description. Also, each addenda has a unique **ID number**. Both these fields can be used for identifying the addenda table in the **Switch Addenda** sequence command as described in Section 6.7.2.

Note: Superfluous addenda tables should periodically be deleted (see Section 6.7.5). A large number of addenda tables may overload the system's memory and impede system performance.

6.7.1 Selecting an Addenda Table Manually

Only one addenda table is active at a time and remains active until a new addenda measurement is performed or a different table is selected. You complete the following steps to select an addenda table:

1. Verify that the correct .cal file is active. Refer to Section 5.7.1. A .cal file contains only the addenda measurements taken for one puck.
2. Open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.

If necessary, adjust the size of the table columns in the **Addenda Tables** dialog box or use the scroll bar to view all information in the columns.

3. Select the correct addenda table as follows: (a) click anywhere on the row that summarizes the data saved in the correct addenda table, (b) click on **Select**, and then (c) click on **OK** in the pop-up message. The entire row is highlighted, and then two arrows move to the left-most column of the row. The addenda table is selected only when the arrows appear to the left of it.
4. Select **OK** to close the **Addenda Tables** dialog box. If necessary, select **OK** to close the **Calorimeter Files** dialog box. The **Addenda** status panel in the Heat Capacity control center now indicates the creation date and temperature range of the addenda table you just selected.

6.7.2 Switching to a New Addenda Table from a Sequence

Switching to a new addenda table under sequence control may be necessary when measuring heat capacity in magnetic fields. Since the heat capacity of the sample holder is somewhat field dependent (see section 6.2.5), automatic addenda switching enables fully automated heat capacity measurements at different fields.

To switch to a new addenda table, simply include the **Switch Addenda** command in your sequence. You can use the command to switch to a new addenda table any one of three different ways.

- **By title.** When selecting by title, you supply a substring contained in the title of the addenda. This is the same title specified in the **Create New Addenda Heat Capacity Table** as shown in Figure 6-5 and in the **Addenda Tables** dialog as shown in Figure 6-9. When more than one addenda table in the current `.cal` file is a match, the most recently acquired table is selected.
- **By magnetic field.** When selecting by magnetic field, the table with the closest magnetic field is selected. Again, in the case of more than one match, the most recently acquired table is selected.
- **By ID number.** When selecting by ID number the specified table is selected. Select the **Browse** button to bring up a list of currently defined addenda tables and associated ID numbers. ID numbers are automatically selected for each addenda table when it is first created.

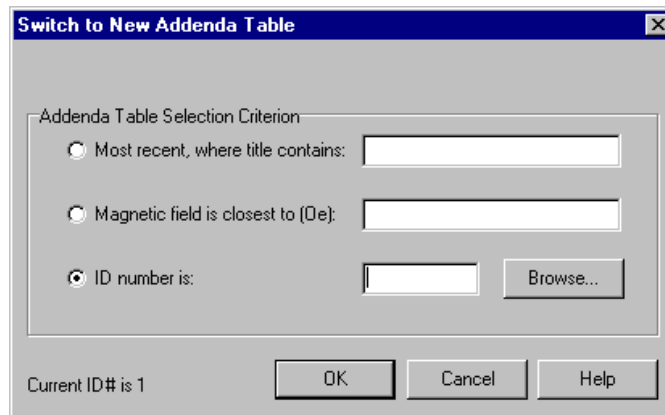


Figure 6-10. **Switch Addenda** Sequence Command Dialog Box

6.7.3 Optional Addenda Offset Tables

In some cases it is useful to subtract a known heat capacity dependence from the one already contained in the addenda table. For example, if your sample is in a container of known mass, it is necessary to subtract the heat capacity of the container to obtain the heat capacity of the contained sample. If the heat capacity of the container has already been measured as a function of temperature, you can simply include that data as part of the addenda.

An offset file must be placed in the `TempCal` directory and must have a `.off` suffix in the file name. The easiest way to create such a file in the proper format is to specify the name of the offset data in the **Advanced** tab of the **Heat Capacity Versus Temperature** dialog box when you are measuring the container. A scale factor can be specified when the table is used.

To specify an offset file for a specific addenda table after the addenda table has been measured, select the **Addenda Offset Formula** button in the **Addenda Tables** dialog box. Then use the **Addenda Offset Formula** dialog box (Figure 6-11) to specify contributions from up to two offset files, each with its own scale factor applied.

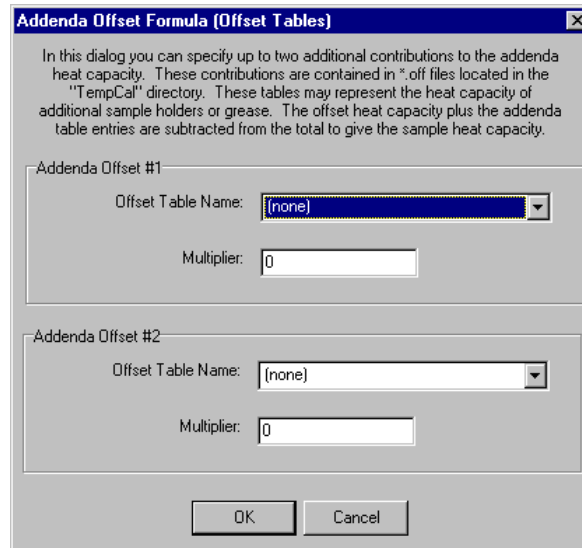


Figure 6-11. Addenda Offset Formula Dialog Box

6.7.4 Editing the Title of an Addenda Table

If a .cal file includes several addenda tables, you should assign descriptive titles to the tables to help distinguish them. You may edit the title of an addenda table at any time; the table does not need to be active.

Complete the following steps to edit the title of an addenda table:

1. Verify that the correct .cal file is active. Refer to Section 5.7.1. A .cal file contains only the addenda measurements taken for one puck.
2. Open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.
3. If you are not editing the title of the active addenda table, click once on the row that is part of the addenda table you want to edit. The row is highlighted, but the two arrows do not move to the left-most row column.
4. Select **Edit Title**. Then edit or enter the title as necessary, and select **OK**. The edits appear in the appropriate **Title** column in the **Addenda Tables** dialog box. If you have edited the title of a nonactive file, the highlight bar returns to the active file.
5. Select **OK** to close the **Addenda Tables** dialog box. If necessary, select **OK** to close the **Calorimeter Files** dialog box.

6.7.5 Deleting an Addenda Table

You may delete any addenda table that is not the active addenda. Once you issue the **Delete** command, the addenda table is deleted permanently and may not be retrieved.

Complete the following steps to delete an addenda table:

1. Verify that the correct `.cal` file is active. Refer to Section 5.7.1. A `.cal` file contains only the addenda measurements taken for one puck.
2. Open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.
3. Click once on any row that is part of a nonactive addenda table. The row is highlighted, but the two arrows do not move to the left-most row column.
4. Select **Delete**, and then select **Yes**. The addenda table is deleted immediately, and the highlight bar returns to the active table.
5. Select **OK** to close the **Addenda Tables** dialog box. If necessary, select **OK** to close the **Calorimeter Files** dialog box.

6.7.6 Advance Addenda Table Manipulation

Under unusual circumstances it may be desirable to edit an addenda table. Clicking the **Addenda Entries** button in the **Addenda Tables** dialog will show the **Calibration Table Edit** dialog for the active addenda table. To change an entry, select the **Edit** tab, change an entry, or paste new entries in the text box following the existing format, then click the **Parse** button. When you are finished, click the **OK** button. To save the addenda, follow the instructions in section 5.7.4 to enable advanced table operations from the **Calorimeter Files** dialog. Then, select the **Advanced >> Save Active Calibration** command. You will also be prompted to save changes to the `.cal` file when you exit the software or select another `.cal` file.

Heat Capacity Measurement

7.1 Introduction

This chapter contains the following information:

- Section 7.2 presents an overview of sample heat capacity measurements.
- Section 7.3 explains how to measure sample heat capacity.
- Section 7.4 explains how to measure sample heat capacity in a magnetic field.
- Section 7.5 describes how to use the thermal cycle command.
- Section 7.6 summarizes and describes the heat capacity measurement parameters.
- Section 7.7 describes the heat capacity measurement process.

7.2 Overview of Heat Capacity Measurement

The Heat Capacity software application calculates the heat capacity of a sample by subtracting the addenda measurement from the total heat capacity measurement. The total heat capacity measurement is the measurement of the heat capacity of the sample, the grease, and the sample platform. The two measurements—one with and one without a sample on the sample platform—are necessary.

For accurate measurement of the sample heat capacity, you first apply a small amount of grease—just enough to hold the sample—to the sample platform, and then you measure the heat capacity of the grease and the platform: This is the addenda measurement, which is discussed in Chapter 6. Next, you mount the sample on the sample platform by pressing the sample onto the grease you have already applied to the platform. You then measure the heat capacity of the sample as described in this chapter. Automatic subtraction of the addenda, interpolated at each sample measurement temperature, is performed.

7.3 Measuring Heat Capacity

7.3.1 Measure the Addenda with Grease

Refer to Chapter 6 to measure the addenda. The addenda heat capacity measurement includes any grease you will use to hold the sample on the sample platform. Section 6.2 presents an overview of the addenda measurement. Section 6.3 explains how to measure the addenda.

If you do not require an accurate measurement of the sample heat capacity, or if you can precisely reproduce the amount of grease used from sample to sample, you may reuse the same addenda table for different samples, rather than remeasure the addenda for each sample. Section 6.7.1 explains how to select an addenda table.

If you are going to perform measurements in magnetic fields, you may need to measure the addenda for each of the fields of interest prior to mounting your sample. See Section 7.4 for more information on measuring heat capacity in a magnetic field.

7.3.2 Prepare the Sample for Measurement

1. Examine the sample to locate its flattest side, and then examine the flattest side of the sample. Look for slight surface irregularities or heavy oxidation, which impede thermal contact between the sample and sample platform.
2. Polish or sand the flattest side of the sample if you notice surface irregularities or oxidation. It is likely that the flattest and cleanest surface of the sample will achieve the best thermal contact with the sample platform.
3. Weigh the sample if you are measuring specific heat or Debye temperature. The software must know the mass of the sample in order to compute specific heat or Debye temperature. Section 1.4.1 discusses sample sizes that Quantum Design recommends you use with the Heat Capacity option.
4. Locate the puck's serial number, which is written on the green fiberglass connector that is at the base of the puck (see Figure 3-2). You need to know the serial number to select the `.cal` file that has been created for the puck.

7.3.3 Mount the Sample on the Sample Platform

1. Plug in the small vacuum pump that is supplied with the sample-mounting station. If the pump has a power switch, turn it on. Otherwise the pump will turn on when you plug it in. Verify that the sample-mounting station is receiving vacuum.
2. Slide the thermal radiation shield off the puck if the shield covers the puck.
3. Place the puck, with the sample platform facing upward, inside the sample-mounting station's puck holder. Rotate the puck until the index key slips into the notch at the rear of the holder, and then *gently* push the puck into the holder. Verify that the sample platform is properly seated on the platform holder.
4. Pivot the puck interlock arm toward the puck so that it grabs and immobilizes the puck. When the interlock arm is against the puck, vacuum pulls the sample platform downward.

A hissing sound in the puck holder indicates a poor seal between the sample platform and holder. To eliminate the leak, remove the puck from the holder, clean off all debris that is under the puck, and then place the puck back inside the holder.

5. Position the sample-mounting station and the puck below a wide field stereo microscope and a strong light, if desired.

CAUTION!

Caution and a steady hand are required to mount and remove samples. You risk breaking the puck wires unless you work very carefully *and* you feel comfortable using tweezers to manipulate samples under the microscope. If you break a wire, you must replace the entire puck frame and platform along with the wires. Section 10.4.1 explains this procedure.

6. Use tweezers to place the sample, with its flattest side downward, on top of the grease that is on the sample platform. Use the blunt edge of a cotton-tipped applicator to *gently* push the sample downward if the sample does not rest flat on the platform. The best thermal contact to the sample occurs when a small amount of grease is squeezed out around all sides of the sample. If you do not see this grease, the surface of the sample is probably not flat or it contains a burr.
7. Push the puck interlock arm away from the puck.
8. Pull the puck off the puck holder. To verify that the sample is attached to the sample platform, you may turn the puck upside down. Be prepared to catch the sample if it is not securely attached to the platform. If the sample falls off, return to step 3.
9. Hold the puck so that the sample platform is facing upward. Slide the thermal radiation shield over the top of the puck, and then twist the shield to verify that it is securely in position. See warning in Section 11.3.6.

Note: Use the puck adjustment tool (see Section 9.2) on the puck after the puck has been inserted in the sample chamber approximately 10 times. By adjusting the tension in the chuck fingers, the puck adjustment tool ensures solid thermal contact between the puck and the heater block located at the bottom of the sample chamber.

10. Use the broken edge of a cotton-tipped applicator to apply a small amount of Apiezon H Grease to each chuck finger (Figure 3-1) if the fingers are dry. Apply only enough H Grease to make the fingers slightly sticky.
11. Unplug the vacuum pump if you will not immediately reuse the sample-mounting station.

7.3.4 Insert the Puck

1. Select the **Installation Wizards** tab in the Heat Capacity control center.
2. Select **Prepare Sample Measurement**. The **Installing Puck for Sample Measurement** dialog box opens.
3. Select **Open Chamber** to vent the sample chamber and warm it to room temperature.
4. Insert the puck into the sample chamber when the on-screen instructions prompt you to do so. The *Physical Property Measurement System: Hardware Manual* discusses puck insertion in detail.

5. Install the baffle assembly. Verify that the charcoal holder is attached to the bottom of the assembly. Refer to Figures 3-7 and 3-8B.

CAUTION!

Do *not* touch the charcoal in the charcoal holder. Touching the charcoal reduces its ability to absorb helium at low temperatures. Touch only the gold plate that surrounds the perimeter of the holder.

6. Insert the baffle assembly into the sample chamber.
7. Select **Purge** to purge and seal the sample chamber.
8. Select **Next**. Then enter the puck's serial number.
9. Select **Next**. The **Current Calibration File** panel opens.

7.3.5 Select a Calibration File

Review the information identifying the active .cal file in the **Current Calibration File** panel (see Figure 7-1). The panel displays summary information—serial number, creation date, name, title, temperature range, and the number of associated addenda tables—for the active .cal file. The serial number identified in the **Current Calibration File** panel should match the serial number you entered previously. A warning message appears at the bottom of the panel if the two numbers do not match.

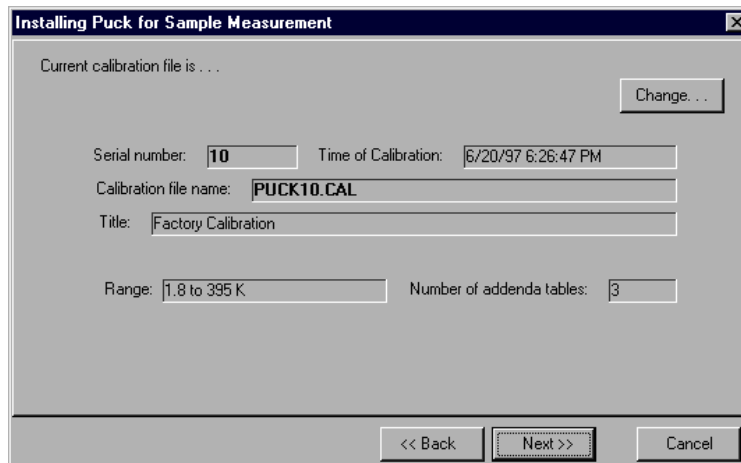


Figure 7-1. **Current Calibration File** Panel in Puck Installation Wizard

- If the serial numbers match, indicating that the correct .cal file is selected, select **Next** to open the **Current Addenda Table** panel.
- If the serial numbers do *not* match, indicating that the correct .cal file is not selected, you must select the correct .cal file. Do the following:
 1. Select **Change** in the **Current Calibration File** panel. The **Calorimeter Files** dialog box opens (see Figure 5-1 or 5-7).

2. Locate the `.cal` file that includes the puck's serial number. The serial number appears in the name of the file and in the **Ser#** column. If necessary, adjust the size of the columns or use the scroll bar to view all information in the columns.
3. Select the correct `.cal` file as follows: (a) click anywhere on the row that summarizes the data saved in the correct `.cal` file, (b) click on **Select**, and (c) click on **OK** in the pop-up message. The file is selected only when the row is highlighted and the two arrows appear to the left of it.
4. Select **OK** in the **Calorimeter Files** dialog box. The **Current Calibration File** panel now displays all data stored in the `.cal` file you have activated. Any warning message disappears if the serial numbers match.
5. Select **Next** to open the **Current Addenda Table** panel.

7.3.6 Select an Addenda Table

Review the information identifying the active addenda table in the **Current Addenda Table** panel (see Figure 7-2). The panel displays summary information—table ID number, creation date, measurement parameters, title, temperature range, magnetic field at which the measurement was obtained, and any additional offsets to be applied—for the active addenda table. A warning message appears at the bottom of the panel if the active table contains no measurement data.

Although you normally want to select an addenda table at this point, it is also possible to select an addenda table from a sequence by executing the **Switch Addenda** sequence command. Refer to Section 6.7.2 for more information on changing addenda tables from a sequence.

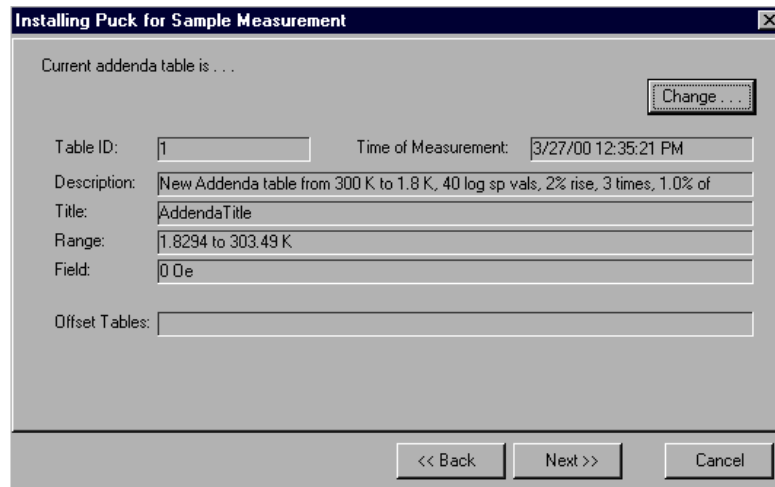


Figure 7-2. **Current Addenda Table** Panel in Puck Installation Wizard

- If you want to use the active addenda table, select **Next** to open the **Puck Test Results** panel.
- If you want to activate another addenda table, do the following:
 1. Select **Change** in the **Current Addenda Table** panel. The **Addenda Tables** dialog box opens (Figure 6-9).
 2. Select an addenda table as follows: (a) click anywhere on the row that summarizes the data saved in the addenda table, (b) click on **Select**, and (c) click on **OK** in the pop-up message. The table is selected only when the row is highlighted and the two arrows appear to the left of it.

3. Select **OK** in the **Addenda Tables** dialog box. The **Current Addenda Table** panel now displays all data stored in the addenda table you have activated.
4. Select **Next** to open the **Puck Test Results** panel.

7.3.7 Test the Puck

The **Puck Test Results** panel displays the results of a functional test of the electrical connections and the resistance of the puck. The Heat Capacity software measures the resistance, at the current temperature, of the platform heater, platform thermometer, and puck thermometer. The test takes a few seconds and begins as soon as the **Puck Test Results** panel opens. When the measurement is complete, a message, indicating the success or failure of the measurement, appears at the bottom of the panel, and the **Measured** and **Expected** temperatures and resistance values appear in the appropriate columns. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

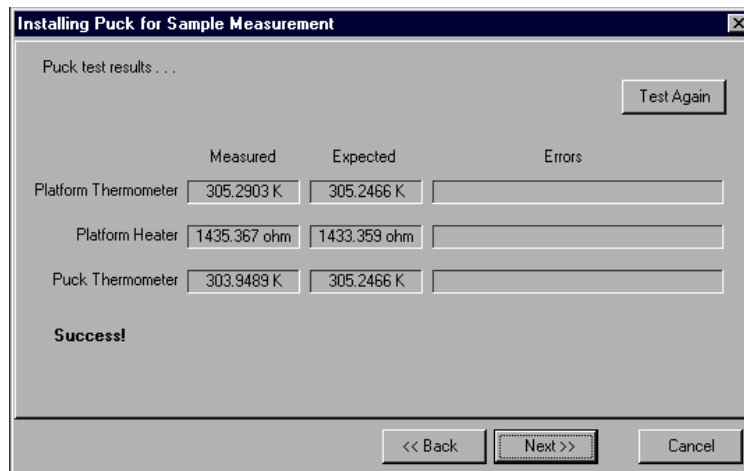


Figure 7-3. **Puck Test Results** panel in puck installation wizard. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

A failure might indicate a broken puck wire or a loose or unplugged cable or that the temperature of the puck is changing too rapidly. If a puck wire is broken, you must replace the puck frame. Refer to Section 9.4.1.

- If you want to retest the puck, select **Test Again** in the **Puck Test Results** panel.
- Select **Next** when you are ready to continue. The **Data File Name** panel opens.

7.3.8 Select a Data File

Note: In certain cases, it might be useful to append new measurements to the end of an existing file. When you append to an existing file, the original header information and the measurement units that were originally selected are extracted from the header of the file. You are not prompted to enter this information when you append to an existing data file.

Review the **Header Information** in the **Data File Name** panel. The **Header Information** lists the information stored in the header of the active data file.

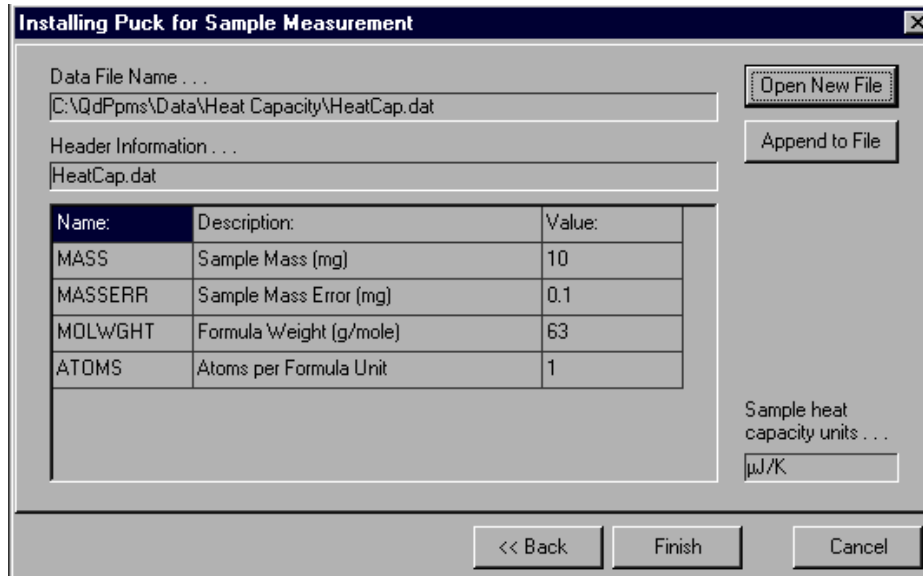


Figure 7-4. **Data File Name** panel in puck installation wizard. The name and location of the active data file are identified at the top of the panel. The information in the header of the active file is displayed below the **Header Information** box. If no data file is active, the **Data File Name** and **Header Information** boxes are blank.

- If you want to use the data file that is currently active, select **Finish**. The **Data File Name** panel closes.
- If you want to append data to a different data file, select **Append to File**, select the file, and then select **Open**. The **Header Information** in the **Data File Name** panel lists the information stored in the header of the file you have just selected. Select **Finish** to close the **Data File Name** panel.
- If you want to create a new data file, do the following:

1. Select **Open New File** in the **Data File Name** panel.
2. Enter the name of the file, and then select **Save**. If you have entered the name of an existing file, a pop-up message asks whether you want to replace the existing file. Select **No**, and then enter another file name.

Once you save the name of the new data file, the **New Data File Information** dialog box opens. The **Optional Information** displayed in this dialog box is the user-defined information that will be saved to the data file header.

3. Enter a different title for the graph view of the file, if necessary. By default, the file name is also the title of the graph view.
4. Review the **Sample Information** that will be saved to the header of the file. Define the **Sample Information** as necessary. Leaving the **Sample Information** fields blank is permissible, but allows the resulting heat capacity measurements to be expressed in only heat capacity units rather than specific heat units. Providing more information increases the choices of specific heat units for expressing the sample data. The mass error is used in calculating the specific heat error during a measurement.

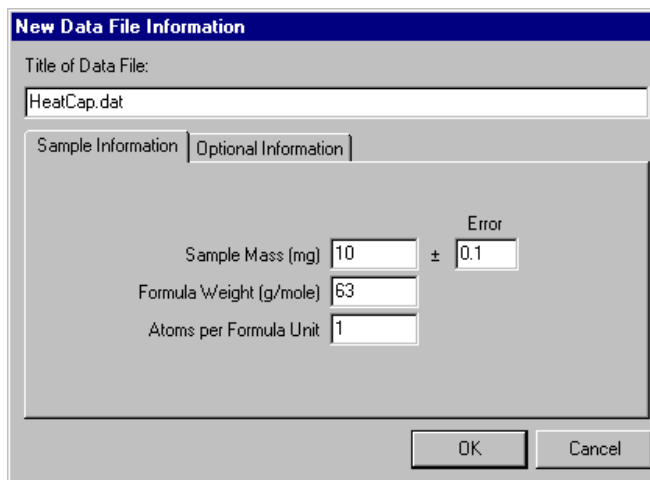


Figure 7-5. Sample information saved to data file header. In this example, all sample information is defined.

5. Review the user-defined **Optional Information** that will be saved to the file header. Select **Configure List** to add, edit, or delete optional items. See Section 4.7.2.1 for more information.
6. Select **OK**. The **Units** dialog box opens if you defined **Sample Information** to be saved in the file header. If you did not define **Sample Information**, the **Data File Name** panel opens again.
7. If the **Units** dialog box is open, select the measurement units, and then select **OK**. The **Data File Name** panel opens again. The **Header Information** now displays the header of the data file you have just opened. The measurement units you have selected appear in the lower right corner.
8. Select **Finish** to close the **Data File Name** panel.

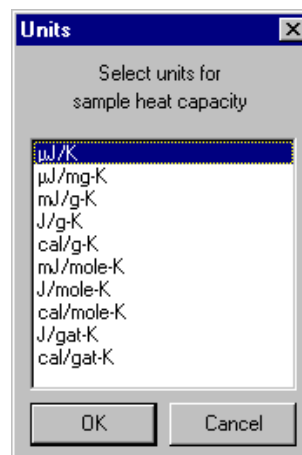


Figure 7-6. Units available for expressing sample data. The number of user-defined sample information fields determines which units are available. In this example, all possible unit selections are available, because all sample information was defined.

7.3.9 Run the Heat Capacity Measurement

1. Select the **Measurement** tab in the Heat Capacity control center.

2. Select **Measure Sample Heat Capacity vs. Temperature**. The **Heat Capacity Versus Temperature** dialog box opens. You use this dialog box to specify the parameters for the sample heat capacity measurement. The dialog box displays the values used for the last sample heat capacity measurement. The parameters are organized into a **Setup** tab (Figure 7-7) and an **Advanced** tab (Figure 7-8). Section **Error! Reference source not found.** explains the parameters in more detail.

CAUTION!

Under certain circumstances, residual helium may be absorbed by components of the calorimeter if the calorimeter is left below 6 K for several hours prior to a measurement. If this is the case, it is recommended that the system be thermally cycled to above 6 K prior to a measurement or that you measure with decreasing temperatures starting above 6 K.

3. Select the **Setup** tab. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

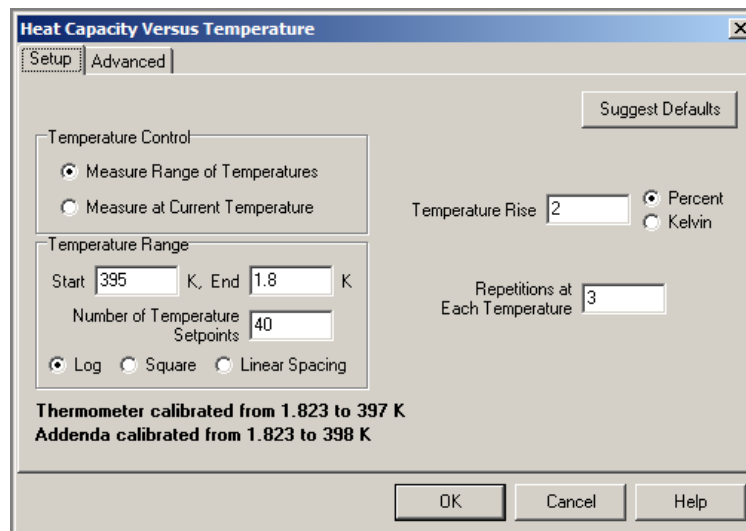


Figure 7-7. **Setup** Tab in **Heat Capacity Versus Temperature** Dialog Box

4. Enter the starting value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range, which are displayed in the lower left corner of the dialog box. The first measurement is taken at the starting temperature value.
5. Enter the ending value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range. The last measurement is taken at the ending temperature value.
6. Enter the number of temperature values, including the starting and ending temperature values, for which you want to take measurements.

Note: Selecting exactly matching measurement points for the sample heat capacity measurement and the addenda measurement is unnecessary. During the sample heat capacity measurement, the software uses polynomial interpolation of the active addenda table at the measurement temperatures for determining the appropriate addenda to subtract.

7. Select the type of spacing that is used to separate the temperature values.
8. Enter a value for the temperature rise.
9. Enter the number of times a measurement is repeated at each temperature.
10. Select the **Advanced** tab if you want to review the advanced measurement parameters. Quantum Design recommends that only default advanced measurement parameters be used. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

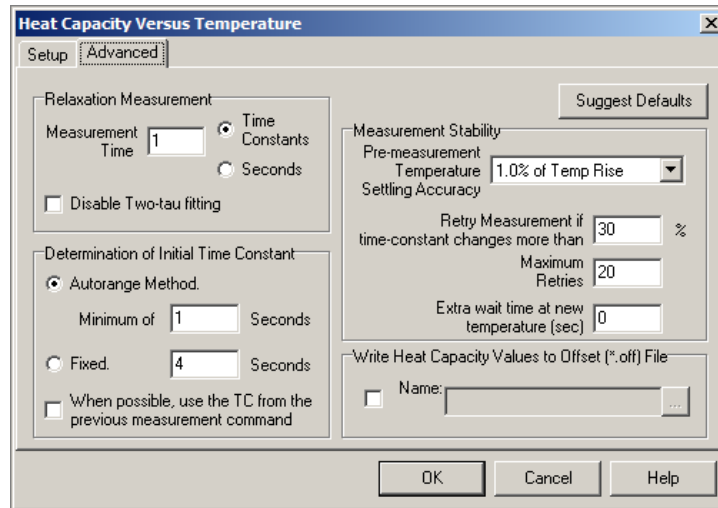


Figure 7-8. **Advanced** Tab in **Heat Capacity Versus Temperature** Dialog Box

11. Select the **OK** button at the bottom of the **Heat Capacity Versus Temperature** dialog box. The sample heat capacity measurement begins if the temperature range you defined is within the thermometer and addenda calibration temperature range.

As soon as the measurement begins to run, the Measurement Status Viewer opens to show the measurement's progress. The name of each task that is part of the measurement appears, as it is performed, in the message list box at the bottom of the Viewer. When the software performs a new measurement, it presents the data in the measurement-field panels and plots the data as a graph. Section 4.5 discusses the Measurement Status Viewer in detail.

The bottom status panel in the Heat Capacity control center also indicates the name of each task as the task is performed.

Section 7.7 describes the heat capacity measurement process in more detail.

12. Wait for the measurement to finish. A measurement lasts several hours or even days. When the measurement is complete, the **Idle** and **Done** messages appear in the bottom status panels in the Heat Capacity control center.

7.3.9.1 PAUSING OR ABORTING A MEASUREMENT

You may pause and resume a sample heat capacity measurement or abort a measurement at any time.

- To pause a measurement, select **Pause** in the Measurement Status Viewer.
- To abort a measurement, select **Abort** in the Measurement Status Viewer. You may abort a paused measurement.

The **Resume** button in the Viewer is enabled when the measurement is paused.

7.3.10 Remove the Puck

Refer to the *Physical Property Measurement System: Hardware Manual* to remove the puck from the sample chamber.

7.3.11 Remove the Sample from the Sample Platform

1. Plug in the small vacuum pump that is supplied with the sample-mounting station. Verify that the sample-mounting station is receiving vacuum.
2. Slide the thermal radiation shield off the puck if the shield covers the puck.
3. Place the puck, with the sample platform facing upward, inside the sample-mounting station's puck holder. Rotate the puck until the index key slips into the notch at the rear of the holder, and then *gently* push the puck into the holder. Verify that the sample platform is properly seated on the platform holder.
4. Pivot the puck interlock arm toward the puck so that it grabs and immobilizes the puck. When the interlock arm is against the puck, vacuum pulls the sample platform downward.
A hissing sound in the puck holder indicates a poor seal between the sample platform and holder. To eliminate the leak, remove the puck from the holder, clean off all debris that is under the puck, and then place the puck back inside the holder.
5. Position the sample-mounting station and the puck below a wide field stereo microscope and a strong light, if desired.

CAUTION!

Caution and a steady hand are required to mount and remove samples. You risk breaking the puck wires unless you work very carefully *and* you feel comfortable using tweezers to manipulate samples under the microscope. If you break a wire, you must replace the entire puck frame and platform along with the wires. Section 10.4.1 explains this procedure.

6. Use tweezers to gently slide the sample off the platform. Never pull the sample directly up off the platform. If you pull the sample directly upward, you may pull the platform off its mount and break the wires.
7. Push the puck interlock arm away from the puck.
8. Pull the puck off the puck holder if you will not immediately mount another sample onto the puck.
9. Unplug the vacuum pump if you will not immediately reuse the sample-mounting station.

7.4 Measurements in a Magnetic Field

Heat capacity can be measured in a magnetic field. However, there are two effects that must be considered before performing such a measurement. First, the sample platform thermometer exhibits a magneto-resistance effect that causes the resistance of the thermometer to change with magnetic field in addition to the normal temperature dependence. The resulting temperature error must be corrected using the calibration procedure described in Chapter 5. In addition, you must be careful to properly account for the magnetic field dependence of the addenda heat capacity. The heat capacity of the platform can depend on the applied field. Hence, before measuring sample heat capacity in a magnetic field, it may be necessary to measure the addenda at each of the desired fields and then select the appropriate addenda, depending on the magnetic field, for the sample measurement. See section 6.2.5 for more information. This procedure should not be necessary for VersaLab systems.

If you are using the Helium-3 insert for performing heat capacity measurements, you may also need to prevent the heat capacity platform from oscillating in the magnetic field during measurements. Due to the orientation of the heat capacity platform on the Helium-3 insert, very small vibrations in the platform may cause large amounts of noise in the measurement. See Chapter 8 for more information on using the Helium-3 insert for measuring heat capacity.

To run a heat capacity measurement in a magnetic field, you complete the following basic steps:

1. Assuming the puck is already calibrated for zero-field operation, perform a secondary field calibration on the same puck at the specific fields at which you will be performing measurements. Refer to Section 5.4. This portion of the calibration should only be necessary from about 20 K to the lowest temperature. Also, it should only be required once for a specific puck.
2. Once the puck is calibrated, prepare for an addenda measurement by applying grease to the platform in the usual way. Then perform an addenda measurement at each of the desired fields as described in Section 6.3. This may not be necessary depending on temperature range, puck type, and accuracy requirements (see Section 6.2.5).
3. Mount your sample and measure the sample heat capacity. At each field, set the field to the desired value. Then select the corresponding addenda table as created above. Finally, measure the sample heat capacity. The **Switch Addenda** command as described in Section 6.7.2 can be used to automatically select the addenda under sequence control.

7.4.1 Example: Measuring Sample Heat Capacity at 0 Oe and 90,000 Oe

It is assumed that the puck has already been calibrated for magneto-resistance at 90,000 Oe.

1. Prepare the addenda by applying a small amount of grease as usual.
2. From the **Installation Wizards** tab press the **Prepare Addenda Measurement** button and follow the instructions to install the calorimeter into the sample chamber, select the appropriate calibration file, test the puck, and specify an optional output file.
3. Prepare a sequence that will perform the following functions:
 - **Set Field** to 90000 Oe
 - **Wait** for Field
 - **New Addenda** from 100 K to 0.5 K
 - **Set Field** to 0 Oe

- **Wait** for Field
 - **New Addenda** from 100 K to 0.5 K
4. Execute the sequence. Note that each addenda command generates another addenda table. Hence after execution is complete, there will be two new addenda tables. The current one will correspond to zero magnetic field since it was the last one acquired. The one at 90000 Oe will be the next one in the list of addenda tables.
 5. From the **Installation Wizards** tab press the **Prepare Sample Measurement** button and follow the instructions to first remove the calorimeter from the sample chamber, mount the sample, install the calorimeter into the sample chamber, select the appropriate calibration file, test the puck, and specify an optional output file.
 6. Once the installation is complete, prepare a sequence that will perform the following functions:
 - **Sample HC** from 100 K to 0.5 K
 - **Set Field** to 90000 Oe
 - **Wait** for Field
 - **Switch Addenda** to the 90000-Oe addenda table
 - **Sample HC** from 100 K to 0.5 K
 7. Execute the sequence. Note that the first command assumes that the zero-field addenda is already selected and that the field is still at zero.

7.5 Thermal Cycle Temperature Command

When measuring heat capacity at low temperatures, it is sometimes useful to warm the sample above a certain critical temperature and cooling it again before performing a heat capacity measurement. For example, for superconducting samples, it might be useful to warm the sample above its critical temperature after changing the magnetic field. Another time when this might be useful is for burning off helium films from porous sample at low temperatures (see Section 11.3.1).

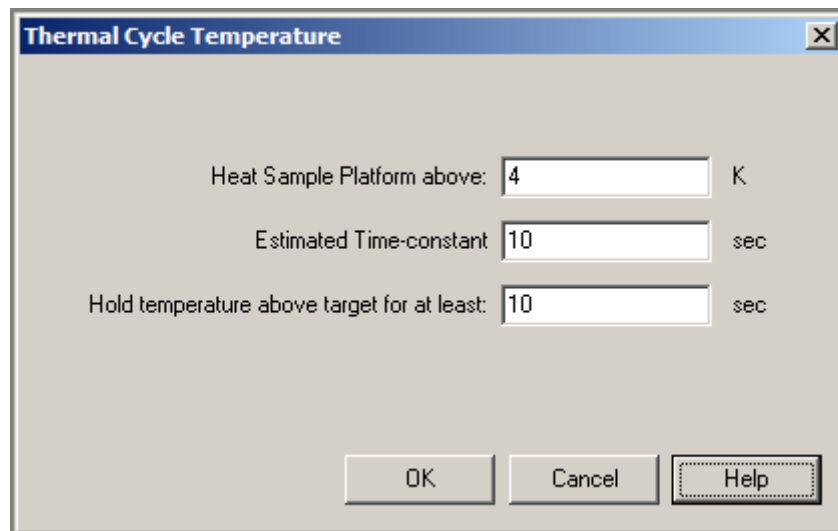


Figure 7-9 Thermal Cycle Temperature Dialog

A sequence command called Thermal Cycle Temperature (Figure 7-9) is provided for warming the platform temperature up to a predetermined temperature and holding it at that temperature for a designated amount of time. While it is possible to cycle the sample temperature using standard temperature control commands, this command is significantly faster since it only warms the sample and sample platform instead of the calorimeter puck and sample chamber. It is particularly convenient when used with the DR insert (Chapter 9) because of the relatively longer time required to change temperatures of the insert. The Thermal Cycle Temperature sequence command also requires that you specify an estimated time constant for more rapid convergence to a temperature that exceeds the target without warming the sample any more than necessary. The maximum temperature that can be reached will vary with temperature and calorimeter, but will always be greater than 20 K.

7.6 Summary of Heat Capacity Parameters

Table 7-1. Parameters in **Setup** Tab in **Heat Capacity Versus Temperature** Dialog Box

PARAMETER	DEFINITION
Temperature Control	How measurement temperature is determined. Select Measure Range of Temperatures to specify a starting and ending temperature. Select Measure at Current Temperature to perform the measurement while stable at the current temperature setpoint. This can be useful for sequence programming where the measurement command appears in a Scan Temperature or Scan Field loop.
Temperature Range Starting Value	Starting value for temperature range. First measurement is taken at this temperature. Heat capacity temperature range should be within temperature range of both thermometer calibration and addenda.
Temperature Range Ending Value	Ending value for temperature range. Last measurement is taken at this temperature. Heat capacity temperature range should be within temperature range of both thermometer calibration and addenda.
Number of Temperature Values	Number of temperature values, including starting and ending value, at which measurements are taken.
Spacing for Temperature Values	Logarithmic, square, or linear spacing between temperature values. Logarithmic spacing equally spaces logarithms of temperature values. Square spacing equally spaces squares of temperature values. Linear spacing equally spaces temperature values.
Temperature Rise	Temperature rise produced by heater-on cycle of heat capacity measurement. Large temperature rise reduces scatter. Small temperature rise is necessary if features such as peaks must be resolved. If temperature rise is too large, it may exceed current limitation of heater driver, or it may produce inaccurate measurement if heat capacity varies substantially over the range produced by the rise. Typical value for temperature rise is 2%. Temperature rise is either given in kelvin or as a percentage of sample temperature at each measurement, depending on user selection. The application automatically determines the heater current needed to produce this temperature rise using the wire conductance tables in the .cal file.
Number of Repetitions Per Measurement	Number of times a measurement is repeated at each temperature. A value of two or more is recommended since this gives a good indication of the self-consistency of measurements.

Table 7-2. Parameters in **Advanced** Tab in **Heat Capacity Versus Temperature** Dialog Box

PARAMETER	DEFINITION
Relaxation Measurement: Measurement Time	<p>Amount of time, including heater-on and heater-off sequences, used for a single relaxation measurement. Measurement time is specified in time constants or seconds. When specified in time constants (the default) the measurement time window is properly scaled for possibly vastly different magnitudes of heat capacity as the temperature changes. When specified in seconds, the measurement time is fixed and does not vary. This setting may help reduce measurement scatter under some circumstances where internal time-constants in the sample cause the reported heat capacity to vary with measurement time.</p> <p>If sample is thermally well attached to the platform, a smaller value for measurement time may reduce scatter. If poorly attached, longer times may be indicated.</p> <p>A typical value for measurement time is 1 time constant. If the puck type is "Standard," the recommended value is 2 time constants for very small samples. This is because the standard puck always uses a measurement time of 2 time constants for addenda measurements.</p>
Relaxation Measurement: Disable Two-tau fitting	<p>When checked, this setting forces the measurement to be fit using the "Simple Model" (single time constant) as described in Section 4.3.3. Under some circumstances, the scatter in the measurement results may be lowered with this setting. Normally, the Two-tau model is used.</p>
Relaxation Measurement: Allow TCs less than Addenda TCs	<p>When checked, this setting allows time – constants for a sample measurement that are less than those for the addenda. Normally, this would be an error condition indicating extremely poor thermal contact between the sample and platform and the system discards such measurements. However, in unusual cases, say when the addenda table has values that make the reported sample heat capacity negative, checking this box will allow the system to report such values.</p>
Determination of Initial Time Constant: Autorange/Fixed Method	<p>This setting governs the determination of the first time constant when the Measurement Time is given in time constants. It only affects the first measurement when a new Heat Capacity Versus Temperature command is executed. When Autorange Method is selected, a trial heat pulse is used. The Minimum of setting makes sure the first measurement is not too short. The Fixed setting is will use the given time as the initial guess. This can save time when you know, within a factor of ten or so, the actual time constant.</p>
Determination of Initial time Constant: When possible, use the TC from the previous measurement command	<p>When checked, this setting will make the initial time constant for this measurement the same as the last measured time constant from previous Heat Capacity Versus Temperature command. This is particularly useful in conjunction with the Measure at Current Temperature setting when the measurement command is part of a Scan Temperature or Scan Field sequence loop. If this is the first measurement since the Heat Capacity option software was activated, the Autorange/Fixed settings are used.</p>
Measurement Stability: Settling Accuracy	<p>Determines how long software waits for temperature stability before taking each measurement. Settling accuracy is given as a percentage of temperature rise. If sample-platform temperature changes between successive readings by less than the settling-accuracy value, temperature is stable enough to proceed with measurement. Small settling accuracies lead to better temperature reproducibility at a specific temperature, though not necessarily better heat capacity values. That is, the temperature scatter will be smaller when this value is smaller but the heat capacity values will still fall on the correct curve. However, the measurement will take longer with a smaller value. A typical value for settling accuracy is 1%.</p>

Measurement Stability: Retry Percentage	Determines how much the time constant is allowed to change from measurement to measurement. For example, a typical value of 30% would mean that if previous relaxation measurement had a time constant of 1 second, and the current measurement was greater than 1.3 or less than 0.7, the current measurement would be discarded and the measurement repeated with the latest acquired time constant. A smaller value may reduce temperature hysteresis effects with some samples by requiring better self-consistency between the assumed time constant used for the "Measurement Time" and the actual time constant determined from the fit.
Measurement Stability: Maximum Retries	Maximum number of times the above condition will discard a measurement before doubling user-specified "Measurement Time." This is an attempt to recover self-consistency. The assumption is that sample is possibly very poorly attached or that specified Measurement Time is much less than 1 and is too short to discern a good value for the time constant when the data is fit. All subsequent measurements will be performed with the longer time constant.
Measurement Stability: Extra wait time at new temperature (sec)	After the PPMS declares stability at a new temperature, the software will wait for the indicated time before starting the measurement. Subsequent measurements at the same temperature do not wait.
Write Heat Capacity Values to Offset File	If this is selected, the heat capacity values obtained from this procedure will be written to the specified <code>.off</code> file in addition to the usual output data file. The offset files reside in the <code>TempCal</code> directory and are used in subsequent measurements as an additional contribution to the addenda. See Section 6.7.3 for more on using these offset tables.

7.7 Description of Heat Capacity Measurement Process

When a sample heat capacity measurement begins, the turbo pump evacuates the sample chamber, and then the chamber temperature stabilizes at the user-defined starting temperature value.

Before the Heat Capacity software can perform the first heat capacity measurement, it must obtain an estimate of the first characteristic heating and cooling time, or *time constant*, of the sample platform. The method used depends on the setting of the **Determination of Initial Time Constant** parameter as described in table 7-2. The **Measurement Time** parameter is expressed in units of time constants, so the software must know the time constant to determine the data acquisition time of the first measurement. Depending on the sample's size and temperature, the time constant could be as small as 100 milliseconds or greater than 100 seconds.

If the **Autorange Method** is chosen, the software reads the active `.cal` file, and by using the value of the thermal conductance of the puck wires, adjusts the temperature of the platform heater in order to create the requested temperature rise. The Heat Capacity Controller uses a timer to monitor the temperature rise until the rise reaches approximately half of its eventual value. The elapsed time that the controller monitors is the initial estimate of the time constant. After the software takes one heat capacity measurement, it estimates the time constant from the measurements. The software usually estimates the initial time constant only once for a given temperature sweep.

Using the estimate of the time constant, the software waits for the platform temperature to stabilize before it takes each measurement. The software monitors the drift rate, which is the slope of temperature versus time, of the platform thermometer at single time constant intervals until it detects no change or detects a change that is less than the change specified by the **Settling Accuracy**

parameter. Once the drift rate is stable, the software applies the heater pulse and records the temperature and heater power, as a function of time, into the Heat Capacity Controller buffer.

The software uses several parameters to fit the time trace of the thermometer response to the two-tau model (see Section 4.3.1.2). This includes an interpolated value for the addenda from the previous addenda measurement.

Before the software takes the next measurement, it appends, as a single record, the relevant data from the fit to the open data file. The software writes the raw data from the measurement and the fit to temporary files in the `Heat Capacity\LogFiles` directory. Section 4.7.4 discusses `.raw` files. Section 4.7.5 discusses `.fit` files.

Operation with the Helium-3 System

8.1 Introduction

This chapter contains the following information:

- Section 8.2 discusses considerations to keep in mind when performing Heat Capacity measurements with the Helium-3 system.
- Section 8.3 explains how to measure the addenda when the Helium-3 insert is installed.
- Section 8.4 explains how to measure the sample heat capacity when the Helium-3 insert is installed.
- Section 8.5 explains how to calibrate the Helium-3 calorimeter puck.

8.2 Special Considerations for Performing Heat Capacity Measurements with the Helium-3 Insert

The PPMS Helium-3 Refrigerator System option (Model P825) and the Heat Capacity option were designed to function together. Performing heat capacity measurements with the Helium-3 system involves the same setup as used without the Helium-3 insert. You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before you proceed with a corresponding measurement using the Helium-3 insert. As with standard pucks, before you measure the heat capacity of a sample, you must first measure the addenda to create a table. Then you can mount the sample on the puck and perform the measurement.

This section describes some of the key differences between heat capacity measurements that are performed with and without the Helium-3 insert. For detailed instructions on calibrating a calorimeter puck for use with the Helium-3 system, refer to Section 8.5.

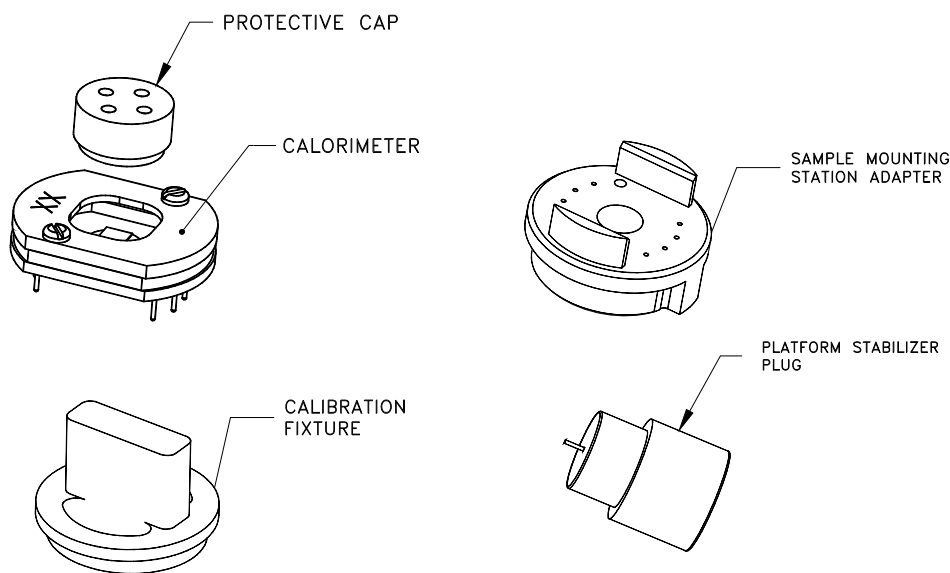


Figure 8-1. Heat Capacity Sample Mount, Mounting Adapter, Calibration Fixture, and Platform Stabilizer

8.2.1 Helium-3 Calorimeter Puck

The calorimeter puck (Figure 8-1) used on the Helium-3 insert is based on the frame assembly used with the standard Heat Capacity puck. The thermometer used on the Heat Capacity platform is specially designed to function at temperatures down to 0.35 K. This thermometer also limits the maximum temperature to 350 K.

Also, because of the difficulty of achieving good thermal contact between materials at these temperatures, the contact wires to the platform have been designed to provide less thermal anchoring to the surroundings. Instead of eight wires for suspending the sample platform, only four wires are used. Furthermore, these wires have approximately half the cross-sectional area of the wires in the standard pucks. This effectively slows down the measurement and thus provides better thermalization of the sample. Unfortunately, this also makes these platforms very delicate. Even slight bumps to the probe when the Heat Capacity puck is mounted can lead to a broken wire.

Due to the construction of the Helium-3 insert, mounting of the Heat Capacity puck differs according to orientation of the sample stage: the puck is mounted vertically in the PPMS for a Helium-3 insert with a vertical sample stage and upside-down for a Helium-3 insert with a horizontal sample stage. Because a mounted sample could slip off the platform in these orientations, a protective cap is provided that twists onto the puck (Figure 8-1). This cap prevents the sample from becoming lost in the unlikely event that the sample slips off. The sample is much more likely to slip off if you are using Apiezon N Grease at temperatures above 300 K. It is recommended that Apiezon H Grease be used at higher temperatures. The cap also serves as protection against putting a finger through the window during mounting and handling.

Another more obvious consequence of the vertical platform orientation is the relationship to the magnetic field. Refer to Section 8.2.2 below.

8.2.2 Measurements in Magnetic Fields

As described in Chapters 5, 6, and 7, the thermometer used on the platform produces temperature errors of up to 50% in magnetic fields of several tesla when used at the lowest temperatures. This magneto-resistance effect requires special handling to perform successful measurements in fields. The Helium-3 system thermometer has a correction built in for correcting temperature errors in the Helium-3 temperature control due to magnetic field. To obtain this correction for heat capacity measurements it is necessary to add magnetic field calibration data to the `.cal` file as described in section 5.4. Also, as described in Chapter 6, the heat capacity of the addenda changes with applied magnetic fields below about 10 K. Successful measurements in magnetic fields below about 2 K will require that an addenda table is measured for each of the corresponding magnetic fields of interest.

8.2.3 Vibration of the Sample Platform

8.2.3.1 VERTICAL SAMPLE-STAGE SYSTEMS

For Helium-3 systems with a vertical sample stage, the electronic measurement of the heat capacity platform thermometer is particularly sensitive to vibrations when a magnetic field is applied. Even the smallest vibrations can produce large disturbances in the measurement data. Such vibrations will appear as an oscillating temperature superimposed on the platform temperature response. The magnitude of this oscillation varies from calorimeter to calorimeter and hence may not be a problem with some pucks.

For this reason, the platform stabilizer (Figures 8-1 and 8-2) has been designed to touch the back of the heat capacity platform with a nylon filament to prevent oscillations without excessive thermal contact. For best results, use it for all heat capacity measurements.

Complete the following steps to mount the platform stabilizer:

1. Mount your sample or prepare your addenda in the usual way and mount the puck onto the Helium-3 insert as described below.
2. If the cap on the puck does not have vent holes incorporated into it, remove the cap from the puck after it is installed. Since the stabilizer plug will block the other hole, the cap is removed so the gas can be evacuated effectively.
3. Remove the protective vinyl cover from the platform stabilizer plug and apply H Grease at the shoulder of the plug as shown in Figure 8-2 on the following page. This grease is used to hold the stabilizer plug in the sample mount.
4. Carefully insert the platform stabilizer into the hole in the backside of the Helium-3 sample stage so that the nylon filament touches the backside of the heat capacity platform. Verify that there is enough grease to hold the plug so that the probe can be inserted without the plug falling out. Only push in the plug far enough to make contact with the platform.

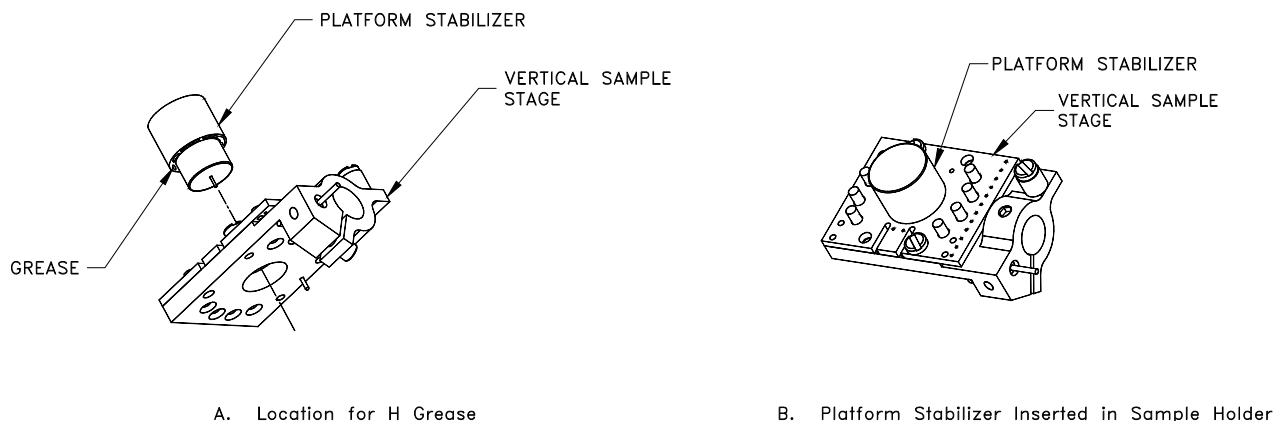


Figure 8-2. Applying H grease to the platform stabilizer and inserting the platform stabilizer into the vertical sample stage.

8.2.3.2 HORIZONTAL SAMPLE-STAGE SYSTEMS

For Helium-3 systems with a horizontal sample stage, vibration in a magnetic field usually does not produce a significant problem. Therefore, the platform stabilizer is not used with Helium-3 systems that have horizontal sample stages. If you experience vibrations with a horizontal platform, it may not affect the resulting measurement data. If it is a problem, please contact Quantum Design.

CAUTION!

The platform stabilizer is not compatible with Helium-3 systems that are equipped with the horizontal sample stage. Attempting to use the platform stabilizer with such systems could result in damage to a heat capacity puck.

8.2.4 Using the Sample-Mounting Station

When you apply grease to the sample platform when you are measuring the addenda or when you are mounting samples, it is necessary to use the sample-mounting station to immobilize the platform. This is particularly critical because of the fragile wires used for support.

A special adapter (part number 4092-625) is provided for the purpose of allowing you to use the standard sample-mounting station with the Helium-3 calorimeter pucks. The following steps describe how you use the sample-mounting station:

1. Remove the calorimeter puck from the Helium-3 insert if the puck is still on the insert.
2. Attach the calorimeter puck to the sample-mounting station adapter (see Figure 8-1). It is a good idea to keep the protective cap on during this step. The eight pins should slip easily into the eight holes and the puck should fit nicely between the two raised sections of the adapter.
3. Use the two long screws through the back side of the adapter to snugly hold the calorimeter onto the adapter. For convenience, coat the screws with H grease before you insert them into the holes in the adapter. The grease keeps the screws from falling out when the puck is not mounted.

Once the two parts are held snugly together, you can start the pump for the sample mounting, insert the assembly onto the mounting station, and completely close the lever arm as you would with a standard puck.

After the sample is mounted or the grease is applied for an addenda measurement, you may remove the assembly from the mounting station in the usual way and remove the calorimeter from the adapter.

8.2.5 Heat Capacity Cable and User Bridge

With a standard Heat Capacity system, the four-pin Lemo on the controller cable (part number 4085-100) is plugged into the system bridge board for reading the puck thermometer. With the Helium-3 system, the Helium-3 thermometer is read by using the standard user bridge board. Therefore, it is necessary to move the cable from the “P2” to the “P1” position on the back of the Model 6000 when going from standard Heat Capacity operations to Helium-3.

8.2.6 Hardware and Software Initialization

The following steps must be completed before performing a Heat Capacity measurement with the Helium-3 system:

1. Review the Helium-3 system operating procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to set up the Helium-3 hardware for a measurement.
2. Start up the Helium-3 software by selecting **Utilities >> Activate Option** in MultiVu and then select and activate **Helium3** in the **Available Options** panel.
3. Minimize the Helium-3 console, if you like. The Helium-3 software runs in the background, so the Helium-3 console does not have to be visible in the MultiVu interface while you work with the Helium-3 system.
4. Start up the Heat Capacity software by selecting **Utilities >> Activate Option** in MultiVu and then select and activate **Heat Capacity for He3**.

8.2.7 Performing Addenda and Sample Measurements

Once the software is running, there are very few procedural differences between running a measurement with a standard puck and a Helium-3 puck. All measurements begin by following the appropriate installation wizard from the Heat Capacity software. Please review the relevant sections of Chapters 6 and 7 for instructions on performing heat capacity measurements.

Other than the lower temperature range and the special handling of the Helium-3 insert—which is described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*—the measurement procedures are identical.

8.2.8 Calibration Files

The calibrations for the Helium-3 calorimeter pucks reside in the `\TempCal\He3` directory. The Helium-3 calibrations are distinguished with names like `He3PuckXX.cal` rather than `PuckXX.cal`, where `XX` is the serial number of the puck. Also, the temperature range for the Helium-3 pucks covers the range from approximately 0.4 to 350 K, whereas the standard pucks cover the range from 1.9 to 400 K.

Section 8.5 discusses the calibration procedure in detail.

8.3 Measuring the Addenda

As with heat capacity measurements without the Helium-3 insert, you must first measure the heat capacity of the platform and grease (addenda) before measuring a sample. Please review the procedures in Chapter 6 before proceeding. Also, it is assumed that you are familiar with the Helium-3 probe-handling procedures described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*.

This section basically repeats the procedures in Chapter 6.

1. Verify that the Heat Capacity DSP cable is properly connected as described in Section 2.3.4.
2. Start the Helium-3 and Heat Capacity software as described in Section 8.2.6.
3. In the **Installation Wizards** tab in the Heat Capacity control center, select **Prepare Addenda Measurement**. Then follow the instructions to warm up and open the sample chamber.
4. Use the sample-mounting station as described in Section 8.2.4 to apply a small amount of grease to the Helium-3 calorimeter platform. As with the standard puck, Apiezon N Grease is preferred, but H Grease can be used if measurements are required above room temperature.
5. Place the protective cap back onto the puck, remove the puck from the mounting station and adapter, and install it onto the bottom end of the Helium-3 insert. Use the .050 hex Allen wrench to gently snug—but not tighten—the two hex Allen screws on the back of the sample-mounting plate to ensure good thermal contact to the mounting surface. Apply a small amount of Apiezon H Grease or Apiezon N Grease at the interface if necessary.
6. Locate and note the serial number on the Helium-3 calorimeter puck. The serial number is written on the top of the puck frame and is needed to start the measurement.
7. Follow the procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to insert the probe into the PPMS sample chamber.
8. Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck.
9. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start an addenda measurement by selecting **Create New Addenda Table** in the **Measurement** tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Refer to Section 6.3.6.

Once the addenda measurement is complete, you may measure a sample by following the procedures in the following section.

8.4 Measuring Sample Heat Capacity

Please review the procedures in Chapter 7 before proceeding. Also, it is assumed that you are familiar with the Helium-3 probe-handling procedures described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*.

This section basically repeats the procedures presented in Chapter 7.

1. Verify that the Heat Capacity DSP cable is properly connected as described in Section 2.3.4.
2. Start the Helium-3 and Heat Capacity software as described in Section 8.2.6.
3. In the **Installation Wizards** tab in the Heat Capacity control center, select **Prepare Sample Measurement**. Then follow the instructions to warm up and open the sample chamber.
4. Use the sample-mounting station as described in Section 8.2.4 to mount a sample onto the grease on the Helium-3 calorimeter platform. The grease should have been applied prior to the addenda measurement.
5. Place the protective cap back onto the puck, remove the puck from the mounting station and adapter, and install it onto the bottom end of the Helium-3 insert. Use the .050 hex Allen wrench to gently snug—but not tighten—the two hex Allen screws on the back of the sample-mounting plate to ensure good thermal contact to the mounting surface. Apply a small amount of Apiezon H Grease or Apiezon N Grease at the interface if necessary.
6. Locate and note the serial number on the Helium-3 calorimeter puck. The serial number is written on the top of the puck frame and is needed to start the measurement.
7. Follow the procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to insert the probe into the PPMS sample chamber. Be very careful not to bump the end of the probe. The extra weight of the sample can easily break wires on the puck if bumped.
8. Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck.
9. Verify that the correct addenda table is selected.
10. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start a sample measurement by selecting **Measure Sample Heat Capacity vs Temperature** in the **Measurement** tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Refer to Section 7.3.9.

8.5 Calibrating the Helium-3 Calorimeter Puck

The calibration of the Helium-3 calorimeter puck, like the calibration of the standard calorimeter puck, consists of two separate parts, or passes. Without the Helium-3 insert, you must first remove the charcoal holder for the first pass of the calibration, and then reinstall it for the second pass. With the Helium-3 insert, you install a calibration fixture (part number 4092-624) for the first pass, and remove it for the second pass. Please review the procedures in Chapter 5 before proceeding.

If you will need to calibrate the thermometers for a specific puck in a magnetic field, refer to Chapter 5 for detailed instructions.

Also, it is assumed that you are familiar with the Helium-3 probe-handling procedures described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*.

8.5.1 Install the Calibration Fixture

To ensure proper thermal equilibrium, you must install the calibration fixture on the calorimeter puck before proceeding.

1. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 8.2.4.
2. Keep the puck on the sample-mounting station and keep the lever closed. Then carefully remove the protective cap from the puck.
3. Apply a small amount of Apiezon N Grease to the tip of the calibration fixture as shown in Figure 8-3. This grease is used to make thermal contact with the sample platform on the puck.

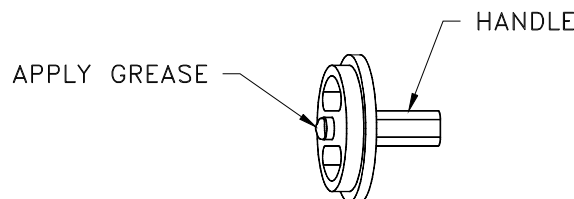


Figure 8-3. Applying Grease to Calibration Fixture

4. Carefully insert the calibration fixture into the window on the calorimeter puck as shown in Figure 8-4. Note that the handle must be oriented diagonally to fit into the window. The tip with the grease should touch the sample platform. Twist the calibration fixture firmly to lock it into place.
5. Remove the puck and adapter from the sample-mounting station.
6. Remove the puck from the black plastic adapter, and then install the puck onto the bottom end of the Helium-3 insert. Do *not* use the handle on the calibration fixture to hold the puck; the fixture can slip out and detach the calorimeter platform.
7. Use the .050 hex Allen wrench to gently snug—but not tighten—the two hex Allen screws on the back of the sample-mounting plate to ensure good thermal contact to the mounting surface. Apply a small amount of Apiezon H Grease or N Grease at the interface if necessary.

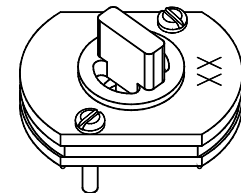


Figure 8-4. Calibration Fixture on Calorimeter Puck

8.5.2 Run the First Pass of the Puck Calibration

As with the calibration process for the standard puck, this pass is used to calibrate the thermometer on the Heat Capacity platform.

1. Verify that the Heat Capacity DSP cable is properly connected as described in Section 2.3.4.
2. Start the Helium-3 and Heat Capacity software as described in Section 8.2.6.
3. In the **Installation Wizards** tab in the Heat Capacity control center, select **Prepare New Puck Calibration**. Then follow the instructions to warm up and open the sample chamber.
4. Locate and note the serial number on the Helium-3 calorimeter puck. The serial number is written on the top surface of the puck and is needed to start the calibration.
5. Follow the procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to insert the probe into the PPMS sample chamber.
6. Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck. Select **He3Puck** as the type of puck you are calibrating.
7. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**. The **Puck Calibration (Pass 1)** dialog box opens.

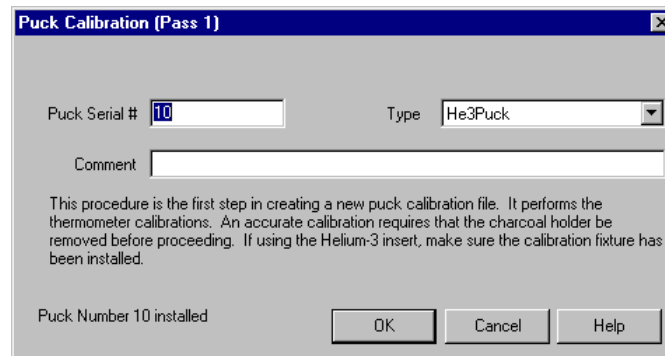


Figure 8-5. **Puck Calibration (Pass 1)** Dialog Box

8. Enter a comment, if desired, then select **OK**. The calibration begins, and the Measurement Status Viewer opens. The length of time the calibration runs varies from system to system. The first pass of the calibration will probably run for approximately 20 hours. The system will then warm up and lead you through the second pass of the calibration.

8.5.3 Remove the Calibration Fixture

Once the first pass is complete, the system warms up and prompts you to remove the calibration fixture. Proceed as follows:

1. Wait for the “Chamber is now flooding” message to appear in the **Setup for Pass 2 of Puck Calibration** dialog box. The message indicates that system conditions are correctly set for removing the Helium-3 insert from the sample chamber. If the system has been idle, you may need to press the **open chamber** button for this.
2. Stand on the step stool included with the Helium-3 option, and then remove the Helium-3 insert from the PPMS sample chamber and place it on the Helium-3 cart. Refer to the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* for detailed instructions.

3. Remove the calorimeter puck from the Helium-3 probe and mount it onto the sample-mounting station as described in Section 8.2.4.
4. Keep the puck on the sample-mounting station and keep the lever closed. Then remove the calibration fixture from the window on the calorimeter puck by twisting to loosen it and *very slowly* raising it out of the window. The chip must be securely held down by the vacuum suction of the mounting station.



5. Once the fixture is removed, place the protective cap back on the puck and remove the puck from the mounting station and adapter.
6. Install the calorimeter puck onto the bottom end of the Helium-3 insert as before.
7. Install the platform stabilizer plug by following the steps in Section 8.2.3.

8.5.4 Run the Second Pass of the Puck Calibration

1. Follow the procedure in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to reinsert the Helium-3 insert back into the sample chamber. If you notice condensation on the probe shaft, you may want to allow the shaft to become warm and dry before continuing. This will speed up the pump-out process.
2. Continue with the instructions in the installation wizard by purging the chamber, performing the puck test, and selecting a new data file, if desired.
3. Select **Finish**. The **Puck Calibration (Pass 2)** dialog box opens.

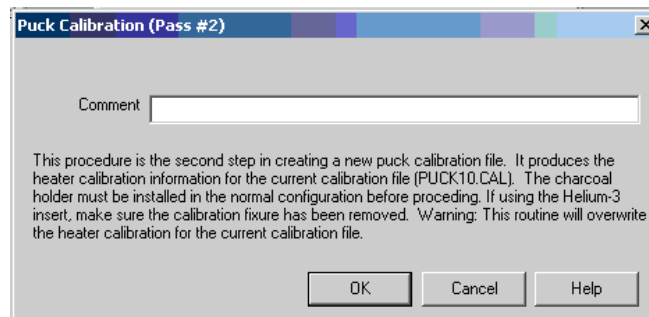


Figure 8-6. **Puck Calibration (Pass 2)** Dialog Box

4. Enter a comment, if desired, then select **OK**. The calibration begins, and the **Measurement Status Viewer** opens. The second pass of the calibration may take more time than the first pass, because when the High-Vacuum system is active, the temperature control is slower. The actual length of time the calibration runs varies from system to system.

When the second pass of the calibration is complete, the system warms up the sample chamber and prompts you to remove the puck. The He3PuckXX.cal file is then complete and ready for measurements.

Operation with the Dilution Refrigerator (DR) System

9.1 Introduction

This chapter contains the following information:

- Section 9.2 describes the special considerations and hardware required for measuring heat capacity with the PPMS DR option.
- Section 9.3 explains how to calibrate the DR calorimeter puck.
- Section 9.4 explains how to measure the addenda with the DR option.
- Section 9.5 explains how to measure the sample heat capacity with the DR option.

9.2 Special Considerations for Performing Heat Capacity Measurements with the Dilution Refrigerator Insert

The PPMS Dilution Refrigerator System (DR) option (Model P850) and the Heat Capacity option were designed to function together. Performing heat capacity measurements with the DR insert is similar to measuring without an insert and is almost identical to measuring with the Helium-3 insert (see Chapter 8). With the DR, heat capacity measurements can be performed over a temperature range from 0.05 K to 4 K, and at the highest fields available with a PPMS.

You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before you proceed with a corresponding measurement using the DR insert. As with standard pucks, before you measure the heat capacity of a sample, you must first measure the addenda to create a table. Then you can mount the sample on the puck and perform the measurement.

This section describes some of the key differences between heat capacity measurements that are performed with and without the DR insert.

9.2.1 Physics of Measuring Heat Capacity at Millikelvin Temperatures

The physics of measuring heat capacity at millikelvin temperatures is the same as that described in Chapter 1 of this manual. However, since the thermal conductivities and heat capacities of matter can be vastly different at 0.05 K than at 1 K, there are significant differences in construction details of a DR calorimeter as compared to the Helium-3 and Standard calorimeters.

Two physical effects are largely responsible for these differences. First, at millikelvin temperatures, there is the so-called Kapitza boundary resistance, where the thermal conductance at the interface between different materials is proportional to the temperature cubed. For example, for a metallic sample greased to a sapphire platform using N-grease, the thermal conductance between sample and the sapphire is approximately 20 microwatts per kelvin at 1 K, yet is only about 10 nanowatts per kelvin at 0.05 K. Because a successful heat capacity measurement requires a certain amount of thermal equilibrium between a sample and the thermometer and heater, this places constraints on the thermal conductance of the thermal link of the calorimeter.

The other important consideration is related to the large disparity between the heat capacities of different materials near 0.05 K and also the effect of magnetic field on heat capacity. Table 9-1 illustrates the enormous variety of heat capacities that different materials can exhibit at, for example, 0.1 K. Note that for a 10 mg sample, the heat capacity of different materials can vary by more than seven orders of magnitude. This representative list puts limits on the types and sizes of samples that can be measured and determines the permissible addenda heat capacity that is acceptable for a useful calorimeter.

Table 9-1 Examples of different types of specific heat in 10 mg samples at 0.1 K.

Physical Source of Heat Capacity	Functional Form	Example Heat Capacity at 0.1 K
Phonons	$C(T) \sim \beta T^3$	10 mg Sapphire: 0.001 nJ/K (extrap)
Electrons	$C(T) \sim \gamma T$	10 mg Gold: 3.5 nJ/K 10 mg Palladium: 88 nJ/K 10 mg Aluminum (normal, ~200 Oe): 50 nJ/K
Heavy Fermions	$C(T) \sim \gamma T$	10 mg PrInAg2: ~15000 nJ/K
Structural tunneling transitions in disordered materials	$C(T) \sim aT^n$ ($1 < n < 2$)	10 mg Apiezon N grease: 1.6 nJ/K 10 mg Stycast 1266 epoxy: 3.1 nJ/K
Magnetic specific heat (Schottky Effect)	$C(T) = C_{\text{schottky}}(T, B)$	10 mg Manganin (Cu-13%Mn): ~10000 nJ/K

Variation of heat capacity with magnetic field can result from both nuclear and electronic energy level splitting in a magnetic field. The resulting Schottky heat capacity anomaly can cause the heat capacity of many materials to vary over several orders of magnitude as the field is changed. If the calorimeter frame is made of materials exhibiting a Schottky effect, the temperature control of the DR sample stage may be adversely affected in a magnetic field. Also, to avoid large variations in the heat capacity

of the platform, all the construction materials of the platform and wires must be chosen carefully to avoid excess Schottky heat capacity.

9.2.2 DR Calorimeter Puck

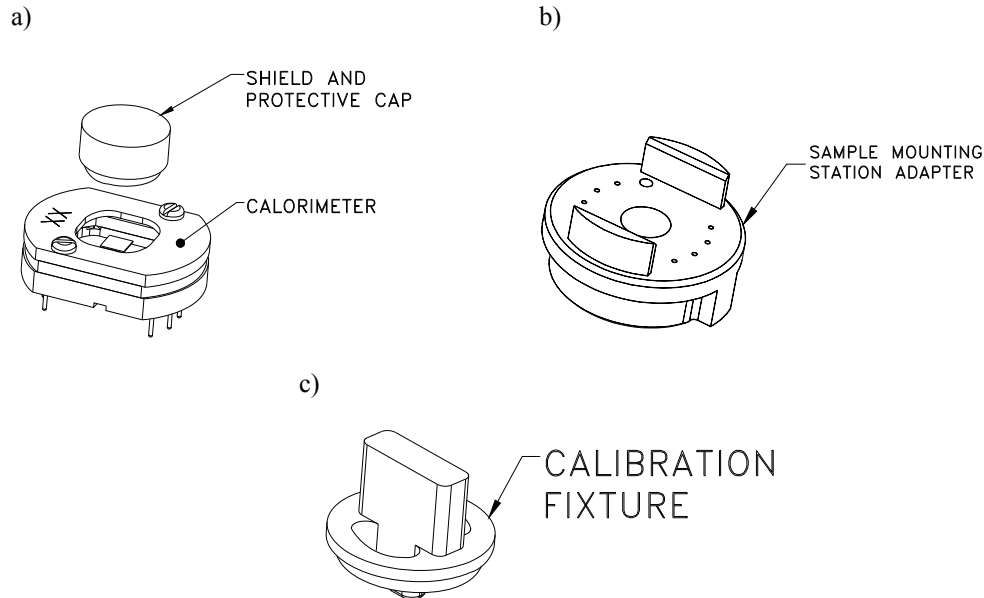


Figure 9-1. (a) Dilution Refrigerator Calorimeter puck (part number 4085-264), (b) Mounting Adapter (4092-625, and (c) Calibration Fixture (4091-624).

The calorimeter puck used on the DR insert is shown in Figure 9-1(a). It is similar in appearance to the frame assembly used with the standard Heat Capacity puck and the Helium-3 puck. The electrical connections and pin layout are identical and there is a locking cap like the Helium-3 puck. Also, the DR calorimeter adapts to the standard sample mounting station using the same adapter used by the Helium-3 calorimeter. However, because of the physics of thermal measurements at 0.05 K, there are also several significant differences between the DR calorimeter and the standard and Helium-3 designs.

9.2.2.1 USING SILVER INSTEAD OF COPPER

The metal frame of the DR calorimeters is made of silver rather than copper. This allows consistent temperature control at high magnetic fields because the heat capacity of silver does not depend on magnetic field down to 0.05 K. The heat capacity of copper at these temperatures, like many materials, has a strong field dependence from the Schottky effect.

Note: Both the DR calorimeter puck and the calibration fixture [Figure 9-1(c)] are made of silver and are then gold plated. With the gold plating, the parts can be easily confused with the corresponding gold-plated copper parts used with the Helium-3 insert (see Figure 8-1). Take precautions to avoid confusion if you also have a Helium-3 insert.

9.2.2.2 THERMAL LINK AND SUSPENSION

The thermal link between the sample platform and the calorimeter frame must be weak enough allow adequate thermal equilibrium to be achieved between the sample and platform during a relaxation measurement. At 0.05 K, the Kapitza boundary resistance limits thermal equilibration between different objects. To achieve reasonable thermal equilibrium between the sample and the platform, the thermal link to the platform should be about a factor of 10 smaller than the sample coupling to the platform. Hence, the thermal link to the bath was designed to be approximately 1 nanowatt per kelvin at 0.05 K. The fine wires needed to achieve this thermal stand-off are inadequate for mechanical support, so polymer supports are used for suspension.

9.2.2.3 THERMAL RADIATION

The frame and cap were also designed with special attention given to screening thermal radiation from outside the puck frame and cap. Because of the low thermal conductance of the thermal link, thermal radiation from the 2 K surroundings can warm the sample platform by several millikelvin. The puck and cap are design to greatly reduce radiative heating by preventing line-of-sight access to the sample platform during normal measurements.

9.2.2.4 THERMOMETER AND HEATER CONSTRUCTION

The resistance thermometer and heater are patterned directly on the underside of the sapphire sample platform. The thick-film materials used for the construction were selected for low Schottky heat capacity. The sensitivity of the thermometer has been tuned for maximum response in the 0.05 K to 4 K range. The calorimeter thermometer is useable up to above 20 K. This is considerably above the 4 K upper limit of the DR insert and can be reached if run on a Helium-3 insert or mounted on a standard puck chuck (see Figure 3-1).

9.2.3 Measurements in Magnetic Fields

As described in Chapters 5, 6, and 7, the thermometer used on the platform produces temperature errors of up to 20% in magnetic fields of several tesla when used at the lowest temperatures. This is also true for the DR platform thermometer. This magneto-resistance effect requires special handling to perform successful measurements in fields. The DR system thermometer has a correction built in for correcting temperature errors in the DR temperature control due to magnetic field. To obtain this correction for heat capacity measurements it is necessary to add magnetic field calibration data to the `.cal` file as described in Section 5.4.

Also, as described in Chapter 6, the heat capacity of the addenda changes with applied magnetic fields. However, depending on how large the heat capacity of your sample is compared to the addenda, it may or may not be necessary to account for this field dependence. In the case where the best possible accuracy is needed, you will need to measure the addenda for each of the corresponding fields of interest.

9.2.4 Vibration of the Sample Platform

Because of the extremely weak thermal link between the platform and calorimeter frame, heat sources as small as 1 pW can create unacceptable levels of heating of the platform. When this heating is not constant in time, the result will be large scatter in the resulting heat capacity measurements. Below about 0.5 K, vibrations of the DR and cryostat can produce substantial vibrational warming of the sample platform. The calorimeter puck has been designed for good rigidity to help address this. However, external vibrations can still produce unacceptable levels of heating on the platform. This may require that the PPMS cryostat be vibrationally isolated from the floor. Please contact Quantum Design for further assistance if you suspect vibrational heating of the calorimeter platform.

9.2.5 Helium Contamination in the Sample Chamber and Thermal Shorting

At DR temperatures, small amounts of helium contamination in the sample chamber can have substantial effects on heat capacity measurements.

9.2.5.1 THE HIGH VACUUM SYSTEM: CRYOPUMPS AND CHARCOAL

The Heat Capacity option always performs measurements under vacuum conditions to minimize thermal conductance through the gas. At DR temperature, there are other effects beside gas conductance that become important, including the heat capacity and thermal conductance of adsorbed helium films. To minimize helium gas and adsorbed superfluid helium films on surfaces in and around the calorimeter puck, the DR software is designed to isolate the sample chamber space from the main chamber cryopump once high vacuum has been achieved. This is normally done at about 20 K during the cooldown of the DR insert. Sealing the chamber prevents back-streaming of helium from the sorption pumps in the cryopump. To remove additional helium from the sample chamber space once it is sealed, there are two additional charcoal sorption pumps located within the sample chamber. One is composed of charcoal cloth and it is mounted on the underside of the screw-on end-cap on the DR insert. This charcoal is maintained at about 1.8 K during normal DR operation. The other charcoal sorption pump is also composed of charcoal cloth and it is located in the underside of the protective cap on the calorimeter puck (see Fig 9-1).

9.2.5.2 SYMPTOMS OF HELIUM CONTAMINATION: ENHANCED HEAT CAPACITY AND THERMAL SHORTING

Samples with a lot of surface area (on a microscopic level) can have a high affinity for helium. In some cases, the result is a slightly larger measured heat capacity. Also, you may find that the measured heat capacity of your sample gets larger over time as more helium adsorbs on it. Another possibility is that the relaxation curves appear to be poorly fit by the thermal model at the lowest temperature.

Another symptom of helium contamination is a Pass 2 calorimeter calibration that fails at or very close to 1.0 K. This failure is caused by an interaction between the polymer supports (small polyimide tubes) described in Section 9.2.2.2 and superfluid helium films. Excess helium in the chamber can create an almost perfect thermal short between the platform and the calorimeter frame. This thermal short appears suddenly below 1 K and gradually goes away again below 0.7 K. So there is no effect above 1.1 K and no effect below 0.5 K. The effect is so dramatic, that it can easily be misdiagnosed as an electronics failure or software bug.

9.2.5.3 CORRECTING HELIUM CONTAMINATION: REGENERATE ALL CHARCOAL

If you suspect helium contamination, you should do the following:

1. Regenerate the sample chamber cryopump. Follow the procedures for your particular system (PPMS or Dynacool).
2. Make sure the charcoal on both the calorimeter cap and the end of the DR insert is fully regenerated. To do this, you will need to remove these two caps and heat them to 150° to 200°C for 1 to 2 hours. This can be done with any oven or hot plate. Before baking, wipe off any excess oils or grease from the metal parts to prevent the contamination from running into the charcoal and clogging the pores. If you suspect that the charcoal in either the calorimeter cap or the DR end-cap has become contaminated with oil, melted grease, or solvents, then the charcoal will need to be replaced. Never bake the entire calorimeter puck.

If you suspect helium contamination from a leak, either in your sample chamber or in the DR insert, please contact Quantum Design for assistance.

9.2.6 Using the Sample-Mounting Station

When setting up a puck calibration, addenda measurement, or sample measurement, it is necessary to use the sample-mounting station to immobilize the platform.

A special adapter [Figure 1(b)] is provided for the purpose of allowing you to use the standard sample-mounting station with the DR calorimeter pucks. The following steps describe how you use the sample-mounting station.

1. Remove the calorimeter puck from the DR insert if the puck is still on the insert.
2. Attach the calorimeter puck to the sample-mounting station adapter. It is a good idea to keep the protective cap on during this step. The eight pins should slip easily into the eight holes and the puck should fit nicely between the two raised sections of the adapter.
3. Optionally, you can use the two long screws through the back side of the adapter to snugly hold the calorimeter onto the adapter. For convenience, coat the screws with H grease before you insert them into the holes to keep the screws from falling out.
4. Insert the assembly onto the mounting station and completely close the lever arm as you would with a standard puck.
5. After the sample is mounted or the grease is applied for an addenda measurement, you may remove the assembly from the mounting station in the usual way and remove the calorimeter from the adapter.

9.2.7 DR Heat Capacity Cable/Filter Box

When the DR is inserted into the PPMS sample chamber for a measurement, the insert is connected to the CM-E Heat Capacity Module using the DR Heat Capacity Cable/RF Filter Box (part number 3091-620), shown in Figure 9-2. This cable assembly has an RF filter box integrated into the end that plugs into the DR insert. The filters eliminate stray RF currents that would otherwise be conducted into the insert from the outside and cause self-heating of the thermometers and anomalous temperature readings. Connect the 15-pin D-shell connector to the CM-E module and connect the filter box to the DR insert at the 10-pin Fischer connector labeled “SAMPLE” (lower connector on the right side of the insert).

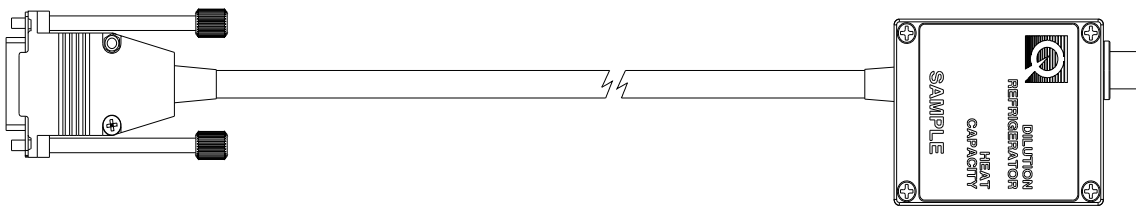


Figure 9-2. DR Heat Capacity Cable/RF Filter Box

9.2.8 Software Initialization

The following steps must be completed before performing a Heat Capacity measurement with the DR system:

1. Review the DR system operating procedures in the PPMS DR User's Manual to set up the DR hardware and make sure the DR gas handling system is connected.
2. Start the MultiVu software if it is not already running and wait for MultiVu to recognize the DR system. This will be indicated by the presence of the DR Option Control Console dialog that pops up shortly after starting MultiVu. Do not connect the TEMPERATURE cable or the SAMPLE cable to the DR insert yet. It is easier to safely handle the DR insert if these connections are made after inserting it into the PPMS sample chamber.
3. Start up the Heat Capacity software by selecting **Utilities >> Activate Option** in PPMS MultiVu and then select and activate **Heat Capacity for DR**. (If the **Heat Capacity for DR** option is not available in the list, you will need to start over and reinstall the Heat Capacity option software as described in Section 2.3.1.)

9.3 Calibrating the DR Calorimeter Puck

The calibration of the DR calorimeter puck, like the calibration of the standard calorimeter puck, consists of two separate parts, or passes. With the standard HC system, you must first remove the charcoal holder for the first pass of the calibration, and then reinstall it for the second pass. With the DR insert, you install the calibration fixture [Figure 9-1(c)] for the first pass, and remove it for the second pass. Please review the procedures in Chapter 5 before proceeding.

To calibrate the puck for measuring in magnetic fields, see the Helium-3 example described in Section 5.4.1.

You only need to follow these calibration procedures once for a given puck. If your DR puck has already been calibrated you may skip this section.

It is assumed that you are familiar with the DR probe-handling procedures described in the PPMS DR System User's Manual.

9.3.1 Install the Calibration Fixture

To ensure proper thermal equilibrium, you must install the calibration fixture [Figure 9-1(c)] on the calorimeter puck before proceeding.

Note: If you also have a Helium-3 inert, do not confuse the calibration fixture described in this section with the one used for Helium-3 calibrations. The one used here is made of silver (see Section 9.2.2.1 above) and is a slightly different shape.

1. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 9.2.5.
2. Carefully remove the radiation cap from the puck and apply a small amount of Apiezon N Grease to the tip of the calibration fixture as shown in Figure 9-3. This grease is used to achieve thermal equilibrium between the fixture and the calorimeter platform.

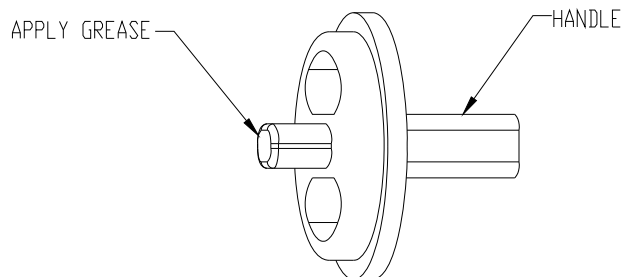


Figure 9-3. Applying Grease to Calibration Fixture

3. Carefully insert the calibration fixture into the window on the calorimeter puck as shown in Figure 9-4. Note that the handle must be oriented diagonally to fit into the window. The tip with the grease should touch the sample platform. Twist the calibration fixture firmly enough to lock it into place.

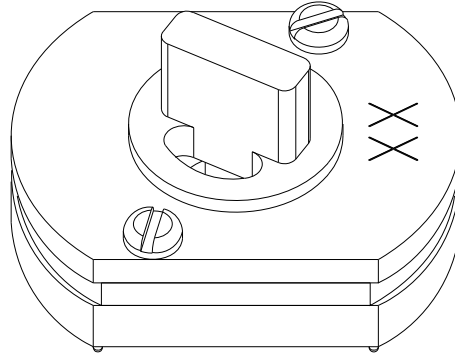


Figure 9-4. Calibration Fixture on Calorimeter Puck

4. Remove the puck and adapter from the sample-mounting station.
5. Remove the puck from the sample-mounting adapter, and follow the instructions in Section 4.4.1 “Mounting and Un-Mounting Samples” in the PPMS DR User’s Manual to attach the calorimeter to the DR insert. Do not use the handle in the calibration fixture to hold the puck; the fixture can slip out and detach the calorimeter platform.
6. Be sure to note the serial number on the calorimeter puck before installing the radiation shield on the end of the DR insert. The serial number is written on the top surface of the puck and is needed to start the calibration.

9.3.2 Run the First Pass of the Puck Calibration

As with the calibration process for the standard puck, this pass is used to calibrate the thermometer on the calorimeter platform with respect to the DR system thermometer.

1. Verify that the software is activated as described above in Sections 9.2.6 and 9.2.7.
2. In the **Installation Wizards** tab in the Heat Capacity control center in MultiVu, select **Prepare New Puck Calibration**. Then follow the instructions to warm and open the sample chamber.
3. Follow the procedures in Section 4.4.2 “Inserting the DR Probe” in the PPMS DR User’s Manual to insert the DR into the PPMS sample chamber. Verify that the Heat Capacity Cable (Figure 9-2) is properly connected to the DR.
4. Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck. Select **DRPuck** as the type of puck you are calibrating.
5. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**. The **Puck Calibration (Pass 1)** dialog box opens.

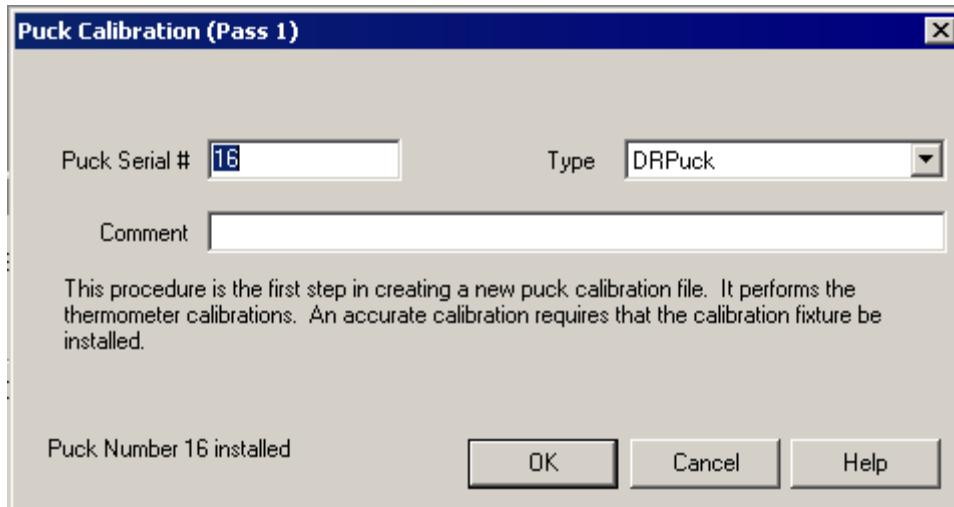


Figure 9-5. **Puck Calibration (Pass 1)** Dialog Box

6. Enter a comment, if desired, then select **OK**. The calibration begins, and the measurement Status View opens. The length of the calibration run varies from system to system. The first pass of the calibration will probably run for approximately 12 hours. The system will then automatically warm up and guide you through the second part of the calibration.

9.3.3 Remove the Calibration Fixture

Once the first pass is complete, the system warms up and prompts you to remove the calibration fixture. Proceed as follows:

1. Wait for the “Chamber is now flooding” message to appear in the **Setup for Pass 2 of Puck Calibration** wizard page. This message indicates that the system is ready for removing the DR insert from the sample chamber.
2. Follow the procedures in Section 4.4.4 “Removing the Dilution Refrigerator Probe” and Section 4.4.5 “Remove the Sample Mount” in the PPMS DR User’s Manual to remove the calorimeter puck from the DR insert.
3. Mount the calorimeter onto the sample mounting station as described in Section 9.2.5. It is critical that the vacuum be applied to hold the platform down during the next step.
4. Keep the puck on the sample-mounting station and keep the lever closed. Then remove the calibration fixture from the window on the calorimeter puck by twisting to loosen it and *very slowly* raising it out of the window. The chip must be securely held down by the vacuum suction of the mounting station.

CAUTION!

If the chip is not securely held down or you try to remove the calibration fixture too quickly, the chip will be pulled off with the fixture and destroyed.

5. Once the fixture is removed, place the calorimeter radiation cap back on the puck and remove the puck from the mounting station and adapter
6. Again, follow the instructions in Section 4.4.1 “Mounting and Un-Mounting Samples” in the PPMS DR User’s Manual to attach the calorimeter to the DR insert.
7. Again, follow the procedures in Section 4.4.2 “Inserting the DR Probe” in the PPMS DR User’s Manual to insert the DR into the PPMS sample chamber and connect the Heat Capacity Cable.

9.3.4 Run the Second Pass of the Puck Calibration

1. Follow the procedures in Section 4.4.2 “Inserting the DR Probe” in the PPMS DR User’s Manual to insert the DR into the PPMS sample chamber. If you notice condensation on the probe shaft, you may want to allow the shaft to become warm and dry before continuing.
2. Continue with the instructions in the installation wizard by purging the chamber, performing the puck test, and verifying the data file.
3. Select Finish. The Puck Calibration (Pass 2) dialog opens.

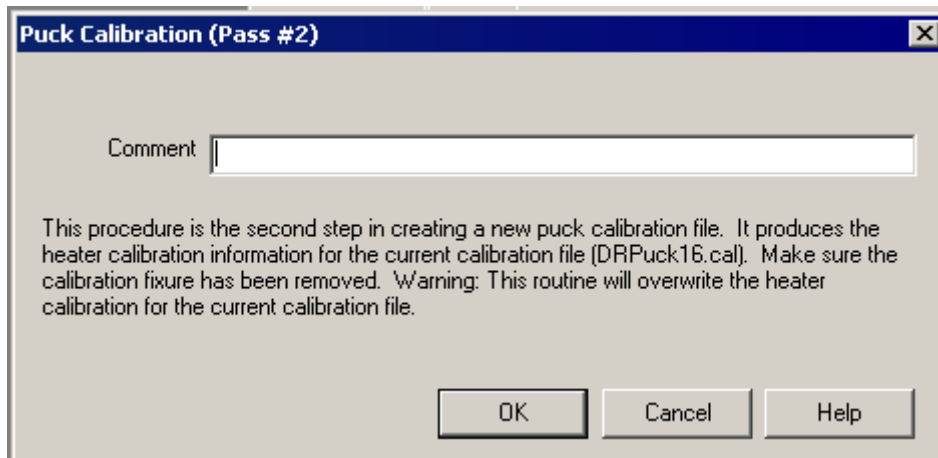


Figure 9-6. **Puck Calibration (Pass 2)** Dialog Box

4. Select **OK**. The calibration begins, and the **Measurement Status Viewer** opens. The second pass of the calibration will take about the same time as the first pass (about 12 hours). The actual length of the calibration will vary from system to system.
5. When the second pass of the calibration is complete, the system automatically warms up the sample chamber and prompts you to remove the puck. The .cal file is selected and ready for measurements.

In addition to calibration data, the pass 2 procedure also creates an addenda table. This addenda table can be used for performing a sample measurement.

9.4 Measuring the Addenda

As with heat capacity measurements without the DR insert, you must first measure the heat capacity of the platform and grease (addenda) before measuring a sample. This procedure is only necessary if there are no previously acquired addenda tables, including the one created during the calibration, that are suitable for your sample. Please review the procedures in Chapter 6 before proceeding. It is assumed that you are familiar with the DR probe-handling procedures described in the PPMS DR System User's Manual.

This section basically repeats the procedures in Chapter 6, modified for the DR insert.

1. Verify that the software is activated as described above in Sections 9.2.6 and 9.2.7.
2. In the **Installation Wizards** tab in the Heat Capacity control center in MultiVu, select **Prepare Addenda Measurement**. Then follow the instructions to warm and open the sample chamber.
3. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 9.2.5.
4. Carefully remove the radiation cap from the puck and apply a small amount of Apiezon N Grease to the DR calorimeter platform.
5. Place the radiation cap back onto the puck, remove the puck and adapter from the sample-mounting station.
6. Remove the puck from the sample-mounting adapter, and follow the instructions in Section 4.4.1 "Mounting and Un-Mounting Samples" in the PPMS DR User's Manual to attach the calorimeter to the DR insert.
7. Be sure to note the serial number on the calorimeter puck before installing the radiation shield on the end of the DR insert.
8. Follow the procedures in Section 4.4.2 "Inserting the DR Probe" in the PPMS DR User's Manual to insert the DR insert into the PPMS sample chamber. Verify that the Heat Capacity Cable (Figure 9-2) is properly connected to the DR.
9. Continue with the instructions in the installation wizard by purging the chamber and verifying the serial number of the puck.
10. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start an addenda measurement by selecting Create New Addenda Table in the Measurement tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Again, refer to Chapter 6.

Once the addenda measurement is complete, you may measure a sample by follow the procedures in the following section.

9.5 Measuring Sample Heat Capacity

Please review the procedures in Chapter 7 before proceeding. Also, it is assumed that you are familiar with the DR probe-handling procedures described in the PPMS DR System User's Manual.

This section basically repeats the procedures in Chapter 7, modified for the DR insert.

1. Verify that the software is activated as described above in Sections 9.2.6 and 9.2.7.
2. In the **Installation Wizards** tab in the Heat Capacity control center in MultiVu, select **Prepare Sample Measurement**. Then follow the instructions to warm and open the sample chamber.
3. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 9.2.5.
4. Carefully remove the radiation cap from the puck and attach the sample to the grease on the calorimeter platform.
5. Place the radiation cap back onto the puck and remove the puck and adapter from the sample-mounting station.
6. Remove the puck from the sample-mounting adapter, and follow the instructions in Section 4.4.1 "Mounting and Un-Mounting Samples" in the PPMS DR User's Manual to attach the calorimeter to the DR insert.
7. Be sure to note the serial number on the calorimeter puck before installing the radiation shield on the end of the DR insert.
8. Follow the procedures in Section 4.4.2 "Inserting the DR Probe" in the PPMS DR User's Manual to insert the DR insert into the PPMS sample chamber. Verify that the Heat Capacity Cable (Figure 9-2) is properly connected to the DR.
9. Continue with the instructions in the installation wizard by purging the chamber and verifying the serial number of the puck, and selecting the correct addenda table. The correct addenda table may already be selected from a previous measurement.
10. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start a sample measurement by selecting **Measure Sample Heat Capacity vs Temperature** in the **Measurement** tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Again, refer to Chapter 7.

Operation with the VersaLab System

10.1 Introduction

This chapter contains the following information:

- Section 10.2 describes the procedures for installing the Heat Capacity Option for the first time.
- Section 10.3 discusses how using the Heat Capacity Option with VersaLab is different from using it on a standard PPMS system.

10.2 Installing the Hardware and Software for VersaLab

This section describes the first-time installation of a new Heat Capacity Option for VersaLab. For standard PPMS systems, follow Section 2.3 instead.

Detailed descriptions of the hardware are contained in Chapter 3. VersaLab-specific hardware is described in this chapter.

10.2.1 Install the Software

The software may already be installed on your PC. If it is not, or if it is an older version, you should obtain installation CDs or download the appropriate installers from the Quantum Design website (www.qdusa.com).

1. Install VersaLab MultiVu version 0.9.17 or higher by running setup.exe from the installation package.

Note: The VersaLab system uses different version numbers for the MultiVu software than for the Standard PPMS.

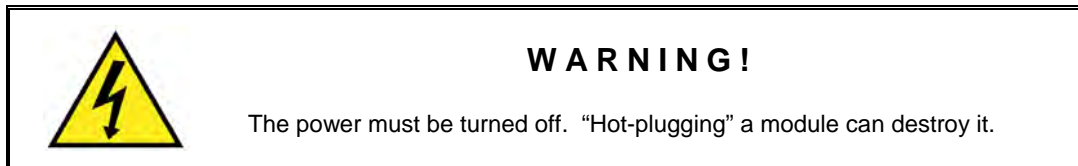
2. Install Heat Capacity version 3.6.0 or later.

10.2.2 Install the CM-E Heat Capacity Controller Module

The control electronics for the Heat Capacity Option are contained in the Model CM-E Heat Capacity Module. See Section 3.2.3 for a drawing and a detailed description of this module. This controller normally resides in one of the integrated module bays on VersaLab and may already have been installed.

If the Heat Capacity Module is not already installed, then do this now:

1. Turn off the power to the VersaLab tower/cryostat.
2. Remove the cover plate from one of the unused module bays and insert the Model CM-E Heat Capacity Module into the empty bay.



3. Once the module is inserted and the mounting thumbscrews screwed in, turn on the power to the VersaLab cryostat. Once the module self-test is complete, the PWR LED will be lit solid green and the COP LED will be solid or blinking green. This indicates normal hardware startup.

10.2.3 Connect the Controller Cable

A single controller cable (3085-110) connects the "Calorimeter JE-1" port on the CM-E Heat Capacity Module to the gray Lemo user port on the cryostat. Figure 10-1 shows this cable. The pin routing for the cable is identical to the standard controller cable and is given in Table 3-1.

Note: The "Puck Therm" (pins 11 thru 14) is not connected for VersaLab.

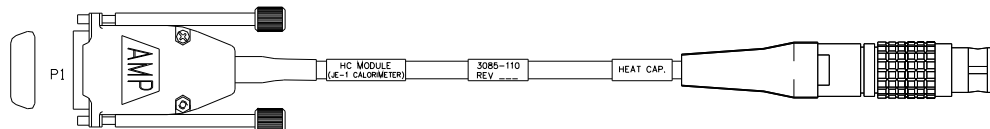


Figure 10-1. Controller Cable for VersaLab Heat Capacity.

10.2.4 Setup Sample Mounting Station

The sample mounting station is a tool to facilitate mounting samples onto the calorimeter puck without damaging the delicate wires suspending the sample platform. For a full description of the mounting station, see Section 3.2.2. To setup the mounting station, follow the instructions in Section 2.3.3.

10.2.5 Quick Start with VersaLab

Once the Heat Capacity Option is installed, follow the instruction in Section 2.4 for quick introduction to performing a first measurement (of addenda) on your VersaLab.

10.3 Special Considerations for Performing Heat Capacity Measurements using VersaLab

The Heat Capacity Option for VersaLab uses the same calorimeter pucks (Section 3.2.1), measurement software (Chapter 4), and measurement electronics (Section 3.2.3) as the standard PPMS Heat Capacity Option. Like the standard PPMS Heat Capacity measurement system, the VersaLab Heat Capacity Option is limited in temperature and field range by the capabilities of the host system. For VersaLab, heat capacity measurements can be performed over a temperature range from 50 to 400 K, and at magnetic fields up to 3 T.

You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before performing Heat Capacity measurements using VersaLab.

This section discusses how using the Heat Capacity Option with VersaLab is different from a using it on a standard PPMS system.

10.3.1 Calorimeter Puck

The VersaLab calorimeter puck is identical to the Standard PPMS Heat Capacity puck as described in Section 3.2.1. In fact, if the puck was calibrated using a model CM-E controller on a standard PPMS, both the puck and calibration file can be used on a VersaLab system.

Other than the limited temperature and field range of the VersaLab as compared to a PPMS, the only difference between the heat capacity options on these system is the use of the puck thermometer in the calorimeter. This auxiliary thermometer, which located inside the puck base, is normally used for faster temperature control. On the VersaLab, there is little or no benefit to using this thermometer, so it is not used. The puck thermometer is included only to maintain compatibility.

10.3.2 High Vacuum and Contact Baffle

With VersaLab, a thermally isolating vacuum is provided in the sample chamber using the built-in cryopump. The contact baffle assembly is included to provide shielding from thermal radiation. This reduces thermal offsets between the calorimeter puck and the chamber block when the chamber is at high vacuum and at either high or low temperatures. The contact baffle is shown in Figure 3-8. For all heat capacity measurements and calibrations, the contact baffle is screwed onto the VersaLab baffle assembly. This baffle assembly is the same as that shown in Figure 3-7, except that it is shorter than the standard PPMS model to match the length of the VersaLab sample chamber.

Since the VersaLab sample chamber does not go below about 50 K, it is not necessary to remove the charcoal assembly from the contact baffle during any part of the puck calibration procedure (see Chapter 5). In fact, the charcoal is not needed at all for high vacuum operations on VersaLab as long as helium exchange gas is used for chamber operations. As a result, unlike with a standard PPMS, both pass 1 and pass 2 of a puck calibration procedure can be done without opening the sample chamber.

10.3.3 Heat Capacity Software

The software used for the VersaLab Heat Capacity Option is the same as other Quantum Design Heat Capacity Options. The installation directory for the VersaLab software is `\QdVersaLab\`. VersaLab-specific installation subdirectories can be found in Table 4-1. For example, calibration files for the VersaLab pucks can be found in the directory `\QdVersaLab\HeatCapacity\TempCal\VersaLab\`. See Chapter 4 for a description of the Heat Capacity software features and options.

Operation with the DynaCool System

11.1 Introduction

This chapter contains the following information:

- Section 11.2 describes the procedures for installing the Heat Capacity Option for the first time.
- Section 11.3 discusses how using the Heat Capacity Option with DynaCool is different from using it on a standard PPMS system.

11.2 Installing the Hardware and Software for DynaCool

This section describes the first-time installation of a new Heat Capacity Option for DynaCool. For standard PPMS systems, follow Section 2.3 instead.

Detailed descriptions of the Heat Capacity hardware are contained in Chapter 3. DynaCool-specific hardware is described in this chapter.

11.2.1 Install the Software

The software may already be installed on your PC. If it is not, or if it is an older version, you should obtain installation CDs or download the appropriate installers from the Quantum Design website (www.qdusa.com).

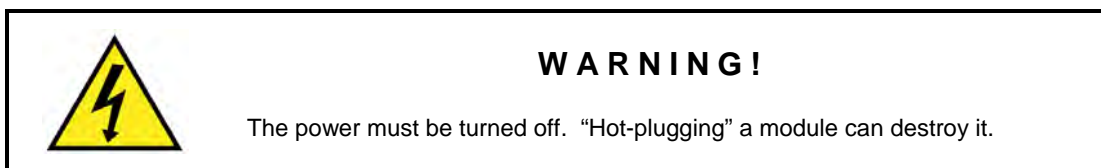
1. Install the latest version of the DynaCool software package by running the installation. This will install the latest version of MultiVu.
2. Install the latest Heat Capacity option software from the Heat Capacity installer.

11.2.2 Install the CM-E Heat Capacity Controller Module

The control electronics for the Heat Capacity Option are contained in the Model CM-E Heat Capacity Module. See Section 3.2.3 for a drawing and a detailed description of this module. This controller normally resides in one of the module bays on the DynaCool CAN Module Bay on the side of the cryostat and may already have been installed.

If the Heat Capacity Module is not already installed, then do this now:

1. Turn off the power to the DynaCool Module Bay and verify that there are no lit indicator lights on any of the installed modules.
2. Remove the cover plate from one of the unused module slots and insert the Model CM-E Heat Capacity Module into the empty slots.



3. Once the module is inserted and the mounting thumbscrews screwed in, turn on the power to the Module Bay. Once the module self-test is complete, the PWR LED will be lit solid green and the COP LED will be solid or blinking green. This indicates normal hardware startup.

11.2.3 Connect the Controller Cable

A single controller cable (3085-110) connects the "Calorimeter JE-1" port on the CM-E Heat Capacity Module to the gray Lemo user port on the right side of the DynaCool cryostat. Figure 11-1 shows this cable. The pin routing for the cable is identical to the standard controller cable and is given in Table 3-1.

Note: The "Puck Therm" (pins 11 thru 14) is not connected for DynaCool.

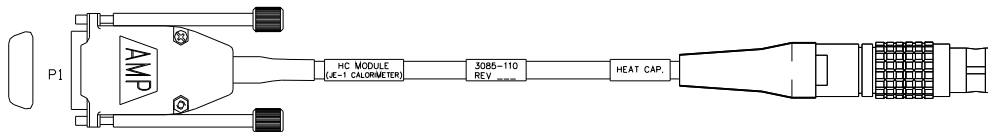


Figure 11-1. Controller Cable for DynaCool Heat Capacity.

11.2.4 Setup Sample Mounting Station

The sample mounting station is a tool to facilitate mounting samples onto the calorimeter puck without damaging the delicate wires suspending the sample platform. For a full description of the mounting station, see Section 3.2.2. To setup the mounting station, follow the instructions in Section 2.3.3.

11.2.5 Quick Start with DynaCool

Once the Heat Capacity Option is installed, follow the instruction in Section 2.4 for quick introduction to performing a first measurement (of addenda) on your DynaCool.

11.3 Special Considerations for Performing Heat Capacity Measurements using DynaCool

The Heat Capacity Option for DynaCool uses the same calorimeter pucks (Section 3.2.1), measurement software (Chapter 4), and measurement electronics (Section 3.2.3) as the standard PPMS Heat Capacity Option. Like the standard PPMS Heat Capacity measurement system, the DynaCool Heat Capacity Option is limited in temperature and field range by the capabilities of the host system. Without any optional refrigerator insert, DynaCool can perform heat capacity measurements over a temperature range from 1.8 to 400 K, and at magnetic fields limited only by your DynaCool magnet.

You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before performing Heat Capacity measurements using DynaCool.

This section discusses how using the Heat Capacity Option with DynaCool is different from a using it on a standard PPMS system.

11.3.1 Calorimeter Puck

The DynaCool calorimeter puck is identical to the Standard PPMS Heat Capacity puck as described in Section 3.2.1. In fact, if the puck was calibrated using a model CM-E controller on a standard PPMS, both the puck and calibration file can be used on a DynaCool system.

The only difference between the heat capacity options on these system is the use of the puck thermometer in the calorimeter. This auxiliary thermometer, which located inside the puck base, is normally used for faster temperature control on a standard PPMS. On the DynaCool, there is little or no benefit to using this thermometer, so it is not used. The puck thermometer is included only to maintain compatibility.

11.3.2 Heat Capacity Software

The software used for the DynaCool Heat Capacity Option is the same as other Quantum Design Heat Capacity Options. The installation directory for the DynaCool software is `\QdDynaCool\`. DynaCool -specific installation subdirectories can be found in Table 4-1. For example, calibration files for the DynaCool pucks can be found in the directory `\QdDynaCool\HeatCapacity\TempCal\Standard\`. See Chapter 4 for a description of the Heat Capacity software features and options.

Hardware Troubleshooting and Maintenance

12.1 Introduction

This chapter contains the following information:

- Section 12.2 explains how to use the puck adjustment tool.
- Section 12.3 explains how to clean pucks.
- Section 12.4 explains how to replace or repair a damaged puck frame.
- Section 12.5 explains how to test the calibration of the user and system thermometers.
- Section 12.6 explains what to do if the vacuum pump for the sample-mounting station produces excess noise.

12.2 Loose Puck: Using the Adjustment Tool

The puck adjustment tool (Figure 12-1) adjusts the tension in the chuck fingers so that the fingers maintain solid thermal contact with the heater block located at the bottom of the sample chamber. Solid thermal contact between the chuck fingers and the heater block is especially important for high-vacuum applications, such as heat capacity measurements.

The puck adjustment tool consists of two metal cylinders. In Figure 12-1, cylinder 1 is the finger spreader, and cylinder 2 is the finger contractor and the test cutout. The finger spreader and the finger contractor adjust the tension of the chuck fingers. The test cutout, which has the same dimensions as the cutout in the heater block, tests how well the chuck fingers will contact the heater block.

You use the puck adjustment tool on the puck after you have inserted the puck into the sample chamber approximately 10 times or whenever the puck fits loosely into the bottom of the sample chamber.

Another indicator of a loose puck is when it takes an unusually long time for the platform temperature to stabilize at a new temperature. For more information on the drift rate, see Section 7.6.

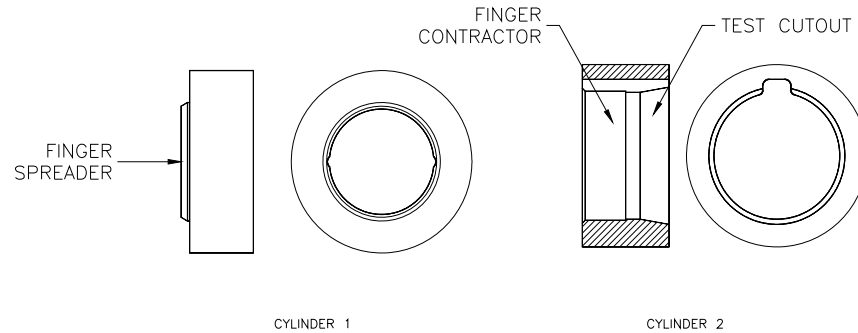


Figure 12-1. Puck Adjustment Tool

Complete the following steps to use the puck adjustment tool:

1. Slide the thermal radiation shield over the top of the puck.
2. Place the puck on the finger spreader. Refer to Figure 12-1. Turn the puck until the screw heads on the bottom of the puck line up with the grooves inside the finger spreader. Press the puck downward and continue pressing until all chuck fingers touch the base of the finger spreader. When all fingers touch the base of the spreader, the spreader evenly applies radial force to the fingers, pushing them outward and slightly beyond their optimal location.
3. Remove the puck from the finger spreader.
4. Place the puck inside the finger contractor. Refer to Figure 12-1. Press straight down on the puck and continue pressing until you press the puck completely into the finger contractor. When the entire chuck is in the contractor, the contractor evenly applies force to the outside of the fingers, pushing them inward. The contractor pushes the fingers—regardless of external wear or variations on the puck—so that the fingers obtain their optimal location.
5. Remove the puck from the finger contractor.
6. Place the puck inside the test cutout. Refer to Figure 12-1. Verify that the puck fits easily but snugly in the test cutout.

12.3 Cleaning Pucks

When you mount samples, you should work carefully to avoid getting grease on the sides or the underside of the sample holder platform. However, when cleaning is necessary, please use the following guidelines.

12.3.1 Degreasing Using a Cotton Swab

To avoid spreading grease on to the sides and underside of the sample platform, it is helpful to routinely remove the grease from the top of the platform after removing a sample and while the puck is still mounted on the sample-mounting station. To remove the grease, gently wipe the surface with a cotton swab moistened with a degreasing agent such as Toluene or 1,1,1-Trichloroethane (TCE). Take care not to touch the wires during this process, as they are very fragile. Also, do not use excessive

amounts of Toluene or TCE because the excess will be pulled into the small vacuum pump, possibly damaging the rubber diaphragms. Finally, avoid leaving behind cotton fibers on the wires or platform.

CAUTION!

Consult the manufacturer's health safety information accompanying both Toluene and TCE. Avoid skin contact and inhalation of vapors.

12.3.2 Degreasing the Entire Frame

If grease has spread to the sides and underside of the sample platform, it may be necessary to degrease the entire frame. Remove the frame from the puck by following step 1 in Section 12.4.1. Submerge the frame in a warm bath of soapy water for 15-30 minutes. Alternatively you can submerge the frame in a room-temperature bath of Toluene or TCE for approximately 5 minutes but no longer than 15 minutes. Rinse the frame in a bath of isopropyl alcohol and let it dry. Follow steps 2 and 3 in Section 12.4.1 to reattach the frame to the puck.

CAUTION!

The puck frame and sample platform are constructed using epoxy. Do not attempt to use any types of solvents not explicitly mentioned in this manual, because they may dissolve the epoxy and permanently damage the frame. Also, do not attempt to use an ultrasonic cleaner.

CAUTION!

This procedure should not be used with DR puck frames. DR calorimeters have internally greased joints that are required for operation. Degreasing agents could remove this grease.

12.4 Broken Puck Wire

A puck is unusable if any of the wires on it breaks. A broken wire is usually indicated by a failed puck test in an installation wizard in the Heat Capacity software.

12.4.1 Replacing the Puck Frame

The Heat Capacity puck is designed so that only part of it needs to be replaced in the event that the puck wires break or the sample platform is damaged. The standard Heat Capacity system is shipped with an additional puck frame for puck repairs. Broken frames can be reworked for a nominal charge, or you can purchase additional spare frames by contacting Quantum Design.

Complete the following steps to replace the puck frame:

1. Remove the two screws located on the bottom of the puck frame, and then remove the puck frame by carefully unplugging it from the eight sockets used for electrical contact. Save the screws.
2. Apply a small amount of Apiezon H Grease to the back of the new frame to ensure adequate thermal contact when the frame is attached to the chuck of the puck. Use just enough grease to make thermal contact, but not so much that an excess amount squeezes out when you assemble the parts.
3. Insert the eight pins of the new frame into the sockets through the top of the body of the puck. The guide pin ensures proper orientation of all parts. Locate the screws you removed in step 1. Use the screws to attach the frame snugly to the puck.
4. Calibrate the puck. Refer to Chapter 5.

12.4.2 Repairing a Damaged Puck Frame

Quantum Design does not recommend that you attempt to repair a broken puck frame. A spare frame is provided to minimize downtime while the broken frame is repaired by Quantum Design personnel. However, if you are compelled to attempt a repair yourself, this section provides some tips. Attempting to repair such a fragile device requires a steady hand and an expertise in soldering fine structures.

For the following procedures, you need tweezers and a soldering iron with a fine tip. You may also need to use a stereo microscope or a lighted magnifying glass.

If a single wire breaks loose from an eight-wire puck frame, you may try a couple different repairs. The simplest thing to do is to short the loose end of the wire to the remaining good wire at the pin rather than at the sample platform. This means that only a three-wire contact is made to the thermometer or heater. A more daring technique is to attempt to solder the wire back into place. Both techniques require that the puck be calibrated again. For these procedures, you need tweezers and a soldering iron with a fine tip.

Complete the following steps to bypass the loose end of the frame to the neighboring pin:

1. Remove the two screws that attach the wire guard to the puck frame, and then remove the wire guard. Save the screws.
2. Place the puck in the sample-mounting station. Turn on the small vacuum pump that is attached to the mounting station and then close the puck interlock arm. Verify that the vacuum holds the sample platform snugly on the platform holder.
3. If your soldering iron has a temperature adjustment, choose the lowest setting that will still melt the solder.

4. Use tweezers to locate the loose end of the broken wire. Solder the loose end to the neighboring pin as shown in Figure 12-2. Care must be taken to ensure that the wire already soldered to that pin remains intact.
5. Place the wire guard on top of the puck frame. Locate the two screws you removed in step 1. Use the screws to attach the wire guard to the puck frame. While you tighten the screws, work carefully so you do not let the wrench slip into the wires.
6. Test the puck by following any of the puck installation wizards in the Heat Capacity software.
7. Calibrate the puck. Refer to Chapter 5.

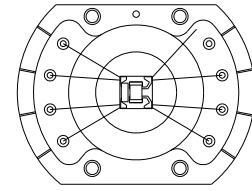


Figure 12.2. Soldering Loose End of Wire to Pin

Connecting the loose end of the broken wire to the pin in this manner is recommended over attempting to reattach the wire directly to the platform. It is very difficult to solder the wire in place and ensure the alignment of the platform when it is subsequently inserted into the sample-mounting station. Moreover, most solders, like SN96, have a superconducting transition at about 3.5 K. There is an anomaly in the heat capacity associated with this transition that will appear as part of the addenda. If only a single wire is repaired with an absolute minimum of solder, it is possible to restrict the size of this bump in the heat capacity to about 1% of the addenda heat capacity at this temperature.

For the adventurous, complete the following steps to reattach the loose end of the wire to the sample platform:

1. Remove the two screws that attach the wire guard to the puck frame, and then remove the wire guard. Save the screws.
2. Remove the two screws located on the bottom of the puck frame, and then remove the puck frame by carefully unplugging it from the eight sockets used for electrical contact. Save the screws.
3. Hold the puck frame so that it is upside down and then place it on a flat surface below a microscope or magnifying glass.
4. Using tweezers and a soldering iron, lightly touch the tinned tip of the iron to bond the wire to the pad. The amount of solder left on the pad should be an absolute minimum due to the anomalous heat capacity contribution to the addenda. The wire wets easily, so very little solder should be necessary to achieve a strong joint.
5. Use the screws to reattach the frame to the body of the puck.
6. Test the alignment of the sample platform by inserting it into the sample-mounting station. The platform is properly aligned if it is pulled snugly onto the platform holder when the vacuum pump is on and the puck interlock arm is engaged. If the platform is properly aligned, go to step 8.
7. If the sample platform does not fit squarely into the platform holder, the length of the repaired wire may have been altered during the repair. To fix this problem, simply touch the soldering iron to the pin (or for certain models, solder pad) to which the repaired wire is attached. While you touch the soldering iron to the pin, you should leave the mounting-station pump running and the puck interlock arm engaged. After touching the iron to the appropriate pin, the stress in the wire will be relieved, thus allowing the platform to slip into the proper position.
8. Place the wire guard on top of the puck frame. Locate the two screws you removed in step 1. Use the screws to attach the wire guard to the puck frame. While you tighten the screws, work carefully so you do not let the wrench slip into the wires.
9. Test the puck by following any of the puck installation wizards in the Heat Capacity software.
10. Calibrate the puck. Refer to Chapter 5.

12.5 Temperatures Read by User Thermometer on Standard PPMS and System Thermometer Do Not Match

If the puck thermometer temperature, as displayed in parentheses on the Model 6000 front panel during a heat capacity measurement, does not match the system thermometer temperature, there may or may not be a problem with the calorimeter puck. The Heat Capacity system operates in vacuum, so a small difference—usually less than 1%—between the puck temperature and the system temperature is likely; this temperature difference is the reason why the puck has its own thermometer. To maintain some moderate thermal contact between the puck and the bottom of the sample chamber, it is important that grease be applied to the puck fingers and that the fingers be periodically spread outward, as described in Section 12.2. To test the calibration of the puck thermometer and the platform thermometer, you complete the following procedure:

1. Perform the **Prepare Addenda Measurement** wizard from the **Installation Wizards** tab in the Heat Capacity control center in order to install the puck and purge the sample chamber as in preparation for measuring addenda.
2. Follow the steps in the wizard until you reach the **Puck Test Results** panel.
3. To verify the calibration at a specific temperature, simply set the new temperature manually from the front panel of the Model 6000 and wait until the temperature is stable.
4. Once the temperature is stable, press the **Test Again** button to compare the **Measured** and **Expected** values for the thermometers.
 - If the temperature difference is greater than 0.5%, the calibration of the puck thermometer is probably in error.
 - If, after performing the preceding test, the calibration is found to be bad, you may need to follow the puck calibration procedures in Chapter 5.
 - If you find that the platform or the puck thermometer is off more than about 1%, you may have a damaged puck. In this case, calibrating it again may help only temporarily or not at all. Please contact your Quantum Design representative if you need additional assistance.

12.6 Sample-Mounting Vacuum Pump Produces Excess Noise

After perhaps 200 hours of operation, the vacuum pump that supplies vacuum to the sample-mounting station may begin to produce a loud noise. Also, the suction of the pump may be reduced. This noise could indicate that the pump's diaphragm is broken. Contact Quantum Design to obtain a replacement diaphragm.

Troubleshooting Heat Capacity Measurements

13.1 Introduction

This chapter contains the following information:

- Section 13.2 discusses indicators of measurement quality in the data file.
- Section 13.3 discusses some common errors that are made during heat capacity measurements.

13.2 Indicators of Measurement Quality in the Data File

When viewing the measurement data contained in a data file, it is important to know whether the data is good or not. Several fields in the data file can be viewed to verify data integrity. While the following sections suggest signs of bad data, the sections do not always give a remedy. Usually the problem can be traced to a sample that is too small or poorly attached to the sample platform. Using nonrecommended values for measured parameters could also be the source of problems.

As a general suggestion, all of the following fields, and many others, can be examined by plotting the field versus Sample Temperature. Often, poor quality measurements are limited to a fraction of the full temperature range of a series of measurements. Sudden changes in a data value with temperature should be investigated carefully.

13.2.1 Heat Capacity Error

If the data file contains data for a sample, you should look at both Samp HC and Samp HC Err. Samp HC Err gives the estimated error contained in the sample heat capacity as given by Samp HC. Samp HC Err corresponds to the error bars. This error is determined by the fitting routine and the quality of the fit as discussed in Chapter 4. If Samp HC Err is more than approximately 10% of Samp HC, you may have a problem.

For an addenda measurement, you should check Addenda HC Err as compared to Addenda HC.

13.2.2 Fit Deviation

The Fit Deviation field contains the normalized chi square for the fit. Where the fit deviation is dominated by thermometer noise this value is approximately 1. Generally, smaller values are better. For most measurements below 200 K, this value is approximately 10. For measurements above 200 K, this value could be as large as 100. If the value is substantially larger than this, the automatic fitting routine may have failed to produce a correct fit, or a glitch occurred during the measurement. The HC Err as described in Section 13.2.1 should reflect this bad fit. Remove the bad data point from the data set if it appears that a spurious glitch occurred.

13.2.3 Sample Coupling

If the Fit Deviation is not too large, the Sample Coupling field indicates how well or how poorly the sample is thermally attached to the sample platform. 100% indicates perfect coupling. The exception is if the fitting algorithm failed to converge to a two-tau fit (see Section 4.3). It is important to distinguish between perfect coupling and a failed fit. For a sample measurement, a failed fit is indicated by the Time Const (tau2) value equal to zero.

If Sample Coupling is less than approximately 90% or the two-tau fit has failed, you might be advised to try to remount your sample and then re-measure it.

Sometimes, certain samples exhibit a large differential thermal contraction with respect to the sapphire calorimeter platform. A dramatic drop in the Sample Coupling (or a failure of the two-tau fit) as the temperature is reduced may indicate a strain between the sample and the platform that results in cracked grease. This is usually only a problem for samples with a large flat surface contacting the calorimeter platform, but can also be a problem for samples with structural transitions. Better result can sometime be obtained by breaking the sample into a number of smaller pieces that are individually attached to the grease on the platform. The theory is that even with the same relative differential strain, the absolute strain will be smaller for each of the pieces, reducing the chance of cracking the grease when the temperature is lowered.

The 90%-rule for Sample Coupling may need to be ignored at temperatures below about 1 K. The reason is that thermal coupling between the sample and the platform may be intrinsically poor at these temperatures (see Section 9.2.1). At these temperatures, a sample coupling of even 70% may be considered acceptable if the fits look reasonably good.

13.2.4 Other Fields to Examine

When you run a heat capacity measurement, you specify a temperature rise in either kelvin or percent. When examining the resulting data, verify that the Temp Rise field is roughly the same as that specified by the measurement parameters. That is, if you specified a Temp Rise of 2% and the Sample Temp field is 6, Temp Rise should equal about 0.12, which is 2% of 6. If it is off by more than approximately 50%, this may be a problem. If the temperature rise is smaller than requested, there may be a problem with the vacuum. This can also be confirmed by examining the Pressure or the Wire Conductance data items.

The Pressure field indicates the pressure at the top of the sample chamber. Normally this will be less than about 0.001 torr. A value larger than this could indicate a vacuum leak or the possible failure of the high vacuum system. Heat capacity measurement acquired under these circumstances should be considered suspect. At temperatures below about 10 K, the Pressure will be of limited diagnostic utility. For example, the charcoal (see Figure 3-8) would absorb Helium leaking into the cold end of

the sample chamber at 2 K. This gas would not be seen by the pressure gauge but would still affect the heat capacity measurement. (See section 13.3.1).

Wire Conductance is computed by the fitting algorithm for both addenda and sample measurements. This field can be a valuable diagnostic for determining the effective gas pressure at the calorimeter. It is especially useful below about 10 K where the pressure gauge may not be sensitive to leaks because of the charcoal. By plotting the Wire Conductance vs Temperature you should verify that the Wire Conductance always increases monotonically with temperature. Also, there should not be significant changes in the Wire Conductance as compared to previous measurements, including the values in the Wire Conductance table stored in the `.cal` file.

When you run a heat capacity measurement, you also specify the measurement time in units of time constants. For example, if you specified 1 for this, the Meas Time field should be approximately equal to the Time Const tau 1 field. If you specified 2, Meas Time should be approximately 2 times larger. Deviations of more than approximately 50% could indicate a problem.

13.3 Common Mistakes and Interesting Results

This section describes common experimental errors associated with performing heat capacity measurements as well as some subtle artifacts specific to the Quantum Design Heat Capacity system. The following list is concerned primarily with effects that not specifically discussed in other sections of this manual. For an updated list of known results, please refer to the Quantum Design website at www.qdusa.com.

13.3.1 Helium Condensation/Adsorption on the Sample or Platform

At temperatures below about 6 K, it is possible to see an anomalous heat capacity due to collection of helium on the platform and in the sample if the sample is particularly porous. This usually appears as a low broad peak around 3 K. The peak will often be worse if the temperature has been below 5 K for an extended period of time. Cycling the temperature up to 20 K will usually temporarily fix the problem (see section 7.5). The charcoal holder on the contact baffle assembly is meant to fix this problem. However, in some cases it can become prematurely saturated. The problem is most likely to appear if the charcoal holder was inadvertently left off or you are measuring a sample that has a greater affinity for helium gas than charcoal.

At DR temperatures, helium contamination can result in superfluid films with dramatic effects including thermal short circuits. See Section 9.2.5 for more information.

13.3.2 Air Leaks and Improper Purging

Another effect related to the helium condensation problem is that of ice contamination of the sample. It is important to purge the sample chamber at temperatures above about 280 K. Even at room temperature, the neck of the PPMS sample chamber can remain cold enough to freeze water and some air constituents. Unless the sample chamber is periodically baked-out at higher temperatures, such that the neck is warmed, there will be a gradual collection of ice on the walls of the sample chamber. If you purge the sample chamber while the puck is cold, the helium gas can eject ice from the walls of the

sample chamber and deposit it on the heat capacity platform. The Heat Capacity system is sensitive enough to detect such contamination. Experimentally, the magnitude of the problem can be seen by repeatedly purging and performing an addenda measurement below, for example, 50 K. With each subsequent purge, the addenda heat capacity will increase slightly. You may even see peaks resulting from an antiferromagnetic transition in frozen oxygen. In rare cases, the air ice may result from a leak in the sample chamber. However, this is usually just a result of poor sample chamber hygiene.

13.3.3 Non-smooth Specific Heat Between 200 K and 300 K due to Anomalous Specific Heat of Apiezon N Grease

Apiezon N Grease exhibits a slight anomaly in its specific heat near its melting point between 260 K and 325 K. This can result in non-smooth results in this temperature range. Please see section 6.2.4.1 and *Quantum Design Heat Capacity Application Note 1085-152A* at www.qdusa.com for more information and specific recommendations.

13.3.4 Scatter in Heat Capacity Data Below 2 K Due to Vibration of Heat Capacity Platform

Below about 2 K, vibrational warming of the heat capacity platform can cause increased scatter in the heat capacity data when using both the Helium-3 insert and the DR insert. Helium-3 systems with vertical sample stages (Section 8.2.3) appear to be the most susceptible to this effect. The platform stabilizer plug (Figure 8-1) can be used to suppress this effect.

With both the Helium-3 insert and the DR insert, some modest vibration isolation of the cryostat from the floor can substantially reduce this effect. Other sources of vibrations come from the PPMS insert itself. This includes oscillations in the gas-flow system and also the turbo pump high-vacuum system (for systems without a cryopump).

A related effect occurs when measuring in a magnetic field where a voltage is induced on the thermometer readback circuit when the calorimeter platform oscillates in the magnetic field. This effect can be identified by an oscillator pattern in the temperature versus time trace in the measurement status viewer.

Contact Quantum Design for help if you are unable to eliminate spurious effects from the vibration sources.

13.3.5 Steps in Heat Capacity vs Temperature

The Heat Capacity control electronics use a set of fixed excitation currents applied to the platform thermometer depending on the temperature. As described in section 5.5, the measured resistance of the platform thermometer can depend slightly on the excitation current because of self-heating effects, especially at lower temperatures. This is why there are different thermometer calibration tables for each excitation current used by the controller. The calibration procedure has been designed to condition these tables to minimize the appearance of a transition in the heat capacity data at temperatures where the excitation current is changed. However, under some circumstances, these steps in the resulting heat capacity vs temperature are still visible. One way to distinguish between these steps which are artifacts of the calibration, and real heat capacity steps, is to examine the Time Const (τ) field versus Sample Temperature. A real step in the heat capacity would also appear in the Time

Const (τ_1) field. If the step was purely due to the calibration, it would appear in the Heat Capacity (or Addenda HC) but not in the Time Const field.

Sometimes these calibration steps result from a flawed calibration run and can be fixed by rerunning the calibration procedure. Also, these steps may result from a short to ground someplace in the calorimeter wiring (probe wiring or on the puck itself), where self-heating is enhanced by unbalancing the impedance of the drive circuit.

13.3.6 Loose Radiation Shield on Standard Puck Causing Elevated and Inconsistent Sample Temperatures

If the lid on the standard calorimeter puck is too loosely attached, and it is cooled down under high-vacuum conditions, the temperature of the lid can lag the temperature of puck by possibly hours and tens of kelvin. Then, thermal radiation from a warmer lid will cause the calorimeter platform (and hence Sample Temperature) to be warmed compared to the System Temperature. Heat capacity measurements acquired under these conditions will occur at higher temperatures than requested, possibly by several kelvin. The amount of heating offset from this effect will become smaller as the lid temperature comes to equilibrium with the puck. When acquiring heat capacity measurements versus temperature, this may appear as an elevated and inconsistent spacing of Sample Temperatures reported in the data file.

The most effective solution to this problem is to make sure that the radiation shield (lid) fits tightly onto the puck frame. See section 3.2.1.2 for information on making the lid fit tightly.

References

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



Vibrating Sample Magnetometer (VSM) Option User's Manual

Part Number 1096-100, B0

Quantum Design

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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
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
Canada 2,089,181 High Symmetry DC Squid System
Japan 2,533,428 High Symmetry DC Squid System
Japan 2,533,428 High Symmetry DC Squid System



Safety Instructions



No operator-serviceable parts are inside. Refer servicing to qualified personnel.



For continued protection against fire hazard, replace fuses only with same type and rating of fuses for selected line voltage.

Observe the following safety guidelines when you use your system:

- To avoid damaging the system, verify that the system power requirements match the alternating current (AC) power available at your location. If the system has not been configured for the correct power available at your location, contact your local service representative before you proceed with the system installation.
- To prevent electrical shock, verify that the equipment is properly grounded with three-wire grounded plugs.
- To prevent electrical shock, unplug the system before you install it, adjust it, or service it.
- Do not spill food or liquids on the system or its cables.
- Refer to the section titled “Safety Precautions” before you install or operate this system. 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 safety eye protection, when you handle cryogenic liquids.
- Transfer liquid helium only in areas that have adequate ventilation and a supply of fresh air. Helium gas can displace the air in a confined space or room, resulting in asphyxiation, dizziness, unconsciousness, or death.
- Keep this system away from radiators and heat sources. Provide adequate ventilation to allow for cooling around the cabinet and computer equipment.
- Refer to the manuals for the supplied computer and monitor for additional safety warnings and notices before you operate the system.

Regulatory Information

- This apparatus has been tested to the requirements of the EMC Directive 89/336/EEC.
- This apparatus is defined as ISM Group 1, Class A and B equipment per EN 50011:1991 (industrial and light industrial environment limits of radio frequency emission).
- This apparatus has been tested to the requirement of the Low Voltage Directive 73/23/EEC.
- See the EU Declaration of Conformity for additional regulatory information regarding your PPMS.

C O N T E N T S

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 provides an overview of the scope of the manual.
- Section P.3 outlines the contents of the manual.
- Section P.4 shows the conventions that appear in the manual.

P.2 Scope of the Manual

This manual contains background about the PPMS Vibrating Sample Magnetometer (VSM) option, as well as instructions for using the VSM software and hardware and performing sensitive measurements when the VSM is installed in the PPMS unit.

P.3 Contents of the Manual

- Chapter 1 provides an overview of the VSM option and the theory of operation and gives contact information for Quantum Design service representatives.
- Chapter 2 describes VSM installation and removal procedures.
- Chapter 3 covers sample preparation and mounting for measurement with the VSM.
- Chapter 4 summarizes the VSM measurement process and guides you through an immediate-mode measurement with the VSM option.
- Chapter 5 describes the hardware and electrical components of the VSM option as well as the VSM User's Kit.
- Chapter 6 describes the VSM application software and the use of immediate-mode and sequence-mode commands to take measurements.

- Chapter 7 describes some troubleshooting strategies for VSM measurements.
- Appendix A provides a functional description of the Model CM-A VSM motor module, including diagrams and electrical specifications.
- Appendix B provides a functional description of the Model CM-B VSM detection module, including diagrams and electrical specifications.
- Appendix C lists Applications and Service Notes pertaining to the VSM Option and provides a place for the user to include new technical notes.

P.4 Conventions in the Manual

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

CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.



WARNING!

Text is set off in this manner to signal conditions that could result in bodily harm or loss of life.



WARNING!

Text is set off in this manner to signal electrical hazards that could result in bodily harm or loss of life.

Introduction to the VSM Option

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the VSM option.
- Section 1.3 discusses the VSM theory of operation.
- Section 1.4 outlines major safety considerations for working with the system.
- Section 1.5 contains information on how to contact your Quantum Design service representative.

1.2 Overview of the VSM Option

1.2.1 What It Measures

The Quantum Design Vibrating Sample Magnetometer (VSM) option for the Physical Property Measurement System (PPMS) family of instruments is a fast and sensitive DC magnetometer.

The basic measurement is accomplished by oscillating the sample near a detection (pickup) coil and synchronously detecting the voltage induced. By using a compact gradiometer pickup coil configuration, a relatively large oscillation amplitude (1–3 mm peak) and a frequency of 40 Hz, the system is able to resolve magnetization changes of less than 10^{-6} emu at a data rate of 1 Hz.

The VSM option consists primarily of a VSM linear motor transport (head) for vibrating the sample, a coilset puck for detection, electronics for driving the linear motor transport and detecting the response from the pickup coils, and a copy of the MultiVu software application for automation and control.

1.2.2 Notable Features of the VSM System

The Quantum Design VSM linear motor transport uses a uniquely designed linear motor to vibrate the sample. Unlike other vibrating sample magnetometers that use a short-throw resonant voice-coil design, you will find that the VSM linear motor is designed to operate at 40 Hz, with

rapid slewing possible over about 6.5 cm of travel. The large range of motion enables the VSM system to perform rapid, completely automated centering operations—you will not need to perform manual adjustments to center the sample.

The sensitivity of the VSM coils is not significantly affected by large magnetic fields, so the VSM can perform sensitive measurements up to the maximum field available from your magnet.

The VSM detection coil is inserted into the sample chamber by using the standard sample interface design. This procedure will make it easy to reconfigure the VSM option with alternate pickup coil designs in the future. You will find that you can change the pickup coil configuration as easily as you can change a puck.

You will find it easy to activate and deactivate the VSM option on your system, just like the other options. The modularity of the design enables you to perform successive types of measurement with little additional effort. For example, you could follow state-of-the-art VSM measurements with heat-capacity measurements by inserting a different puck or probe.

1.3 Theory of Operation

The basic principle of operation for a vibrating sample magnetometer is that a changing magnetic flux will induce a voltage in a pickup coil. The time-dependent induced voltage is given by the following equation:

$$\begin{aligned} V_{coil} &= \frac{d\Phi}{dt} \\ &= \left(\frac{d\Phi}{dz} \right) \left(\frac{dz}{dt} \right) \end{aligned} \tag{1.1}$$

In equation (1.1), Φ is the magnetic flux enclosed by the pickup coil, z is the vertical position of the sample with respect to the coil, and t is time. For a sinusoidally oscillating sample position, the voltage is based on the following equation:

$$V_{coil} = 2\pi f C m A \sin(2\pi f t) \tag{1.2}$$

In equation (1.2), C is a coupling constant, m is the DC magnetic moment of the sample, A is the amplitude of oscillation, and f is the frequency of oscillation.

The acquisition of magnetic moment measurements involves measuring the coefficient of the sinusoidal voltage response from the detection coil. Figure 1-1 illustrates how this is done with the VSM option.

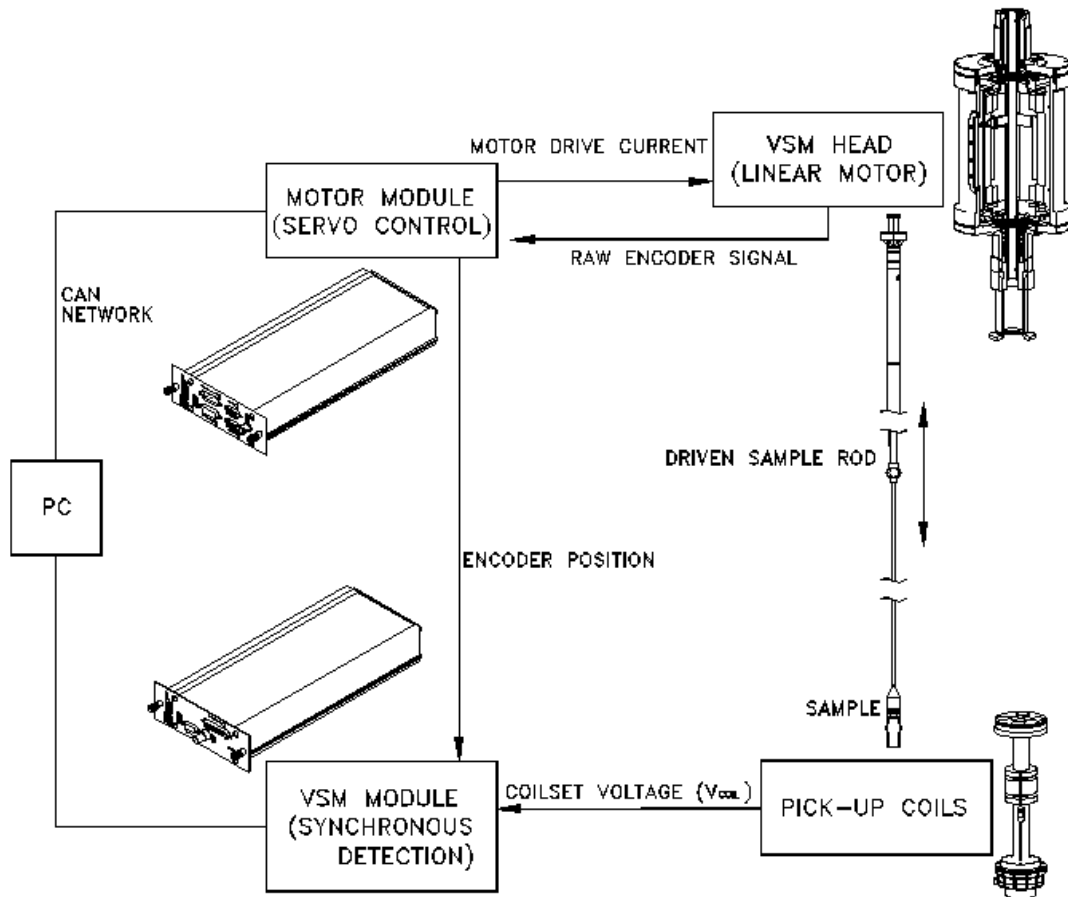



Figure 1-1. Operating principle for the VSM option

The sample is attached to the end of a sample rod that is driven sinusoidally. The center of oscillation is positioned at the vertical center of a gradiometer pickup coil. The precise position and amplitude of oscillation is controlled from the VSM motor module using an optical linear encoder signal readback from the VSM linear motor transport. The voltage induced in the pickup coil is amplified and lock-in detected in the VSM detection module. The VSM detection module uses the position encoder signal as a reference for the synchronous detection. This encoder signal is obtained from the VSM motor module, which interprets the raw encoder signals from the VSM linear motor transport. The VSM detection module detects the in-phase and quadrature-phase signals from the encoder and from the amplified voltage from the pickup coil. These signals are averaged and sent over the CAN bus to the VSM application running on the PC.

Chapter 5 describes the hardware components of the Quantum Design VSM option in more detail.

1.4 Safety Precautions


	WARNING!
<p>The VSM option is used in conjunction with the Physical Property Measurement System (PPMS) family of instruments, so you should be aware of the safety considerations for both pieces of equipment. Potential safety precautions include those for the use of superconducting magnets and for the use of cryogenic liquids (if applicable). Consult your base system User Manual for more specifics.</p>	

Above all, Quantum Design and its staff ask that you use standard safe laboratory procedures.

- ❖ Use common sense.
- ❖ Pay attention to the system's state and your surroundings.
- ❖ If the behavior of the system appears abnormal, something may be wrong with it. Investigate, and if necessary, take appropriate action.
- ❖ Supervise inexperienced users and train them in general electrical safety procedures.

The VSM and PPMS family of instruments have safety features to prevent accidents from causing injury or serious equipment damage. *If you use the equipment in a manner that is not specified by Quantum Design, the protection afforded by the equipment may be impaired.*

1.4.1 Magnets

	WARNING!
<p>Any person who wears a pacemaker, electrical medical device, or metallic implant must stay at least 5 m (16.5 ft.)¹ from the dewar. In addition, personnel should keep all ferromagnetic objects at least 5 m (16.5 ft.) from the dewar. Verify that all magnetic fields are at zero (0) before you handle the VSM linear motor transport in any way.</p>	

The following precautions should be followed to ensure the safety of personnel who work with or around a superconducting magnet. This material is covered in more depth in your system User Manual.

- ❖ Verify that any person who has a metallic implant or is wearing a pacemaker or electrical or mechanical medical device stays at least 5 m (16.5 ft.) from the dewar. Large magnetic fields


¹ At the current time (August 2010), 5 m should be a large enough distance to protect wearers of metallic implants or medical devices from most magnetic fields produced by Quantum Design magnets. However, the safe distance from newer magnets (in development) could be greater. Please consult Application Note 1070-204 on the Quantum Design website www.qdusa.com for detailed information about stray magnetic fields from the magnets in use.

are dangerous to anyone who has a metallic implant or is wearing a pacemaker or other electrical or mechanical medical device.

Important: The automated control system can turn on the magnet while the system is unattended. Furthermore, the three-dimensional magnetic field will penetrate nearby walls, the ceiling, and the floor. Therefore, your safety considerations should include such adjacent spaces.

- ❖ Keep all iron, nickel, and other ferromagnetic objects at least 5 m (16.5 ft.) from the dewar. Large magnets, such as superconducting magnets, can attract iron and other ferromagnetic materials with great force.
- ❖ Never attempt to install, remove, or handle the VSM linear motor transport (4096-400) when there is a field set in the system or in any other nearby equipment. In addition, the VSM linear motor transport must be secured when it is stored within 5 m (16.5 ft.) of the system or any other large field source. The VSM linear motor transport contains nearly 9 kg of iron, which presents a considerable hazard in a large magnetic field such as that produced by the system or other laboratory equipment such as an NMR magnet.

1.4.2 Cryogenics (if applicable)

WARNING!	
	Always wear protective clothing and ensure that the room has good ventilation when you work with cryogenic materials such as liquid helium and liquid nitrogen. These precautions will protect you against cryogenic material hazards: (1) they can expand explosively when exposed to room temperature; (2) they can cause serious burns.

- ❖ Always wear protective clothing, including thermal gloves, eye protection, and covered shoes, when you work with liquid helium, liquid nitrogen, or other cryogenics. Avoid loose clothing or loose fitting gloves that could collect cryogenic liquids next to the skin. The extreme cold of liquid and gaseous cryogenics can cause serious burns and has the potential to cause loss of limbs.
- ❖ Work with cryogenic materials in well-ventilated areas only. In the event a helium container ruptures or there is a helium spill, vent the room immediately and evacuate all personnel. In a poorly ventilated area, helium can displace the air, leading to asphyxiation. Because helium rises, well-vented rooms with high ceilings generally provide the safest setting for working with helium.

1.4.3 Electricity



WARNING!

The VSM and the PPMS family of instruments are powered by nominal voltages that range from 100 V to 240 V AC. These voltages are potentially lethal, so you should exercise appropriate care before opening any of the electronics units, including turning off the equipment and disconnecting it from its power source.

- ❖ Turn off and unplug all electronic equipment before removing any equipment covers.
- ❖ Keep electrical cords in good working condition and replace frayed and damaged cords.
- ❖ Keep liquids away from the workstations.

1.4.4 Lifting and Handling

The VSM linear motor transport (4096-400) should be handled with care, as it is very heavy (about 10 kg or 22 lb) and could cause crushing injuries.

1.5 Contacting Quantum Design

If you have trouble with your VSM or your system, please contact your local Quantum Design service representative for assistance. See www.qd-international.com for the information about your local representative. You will be asked to describe the problem, the circumstances involved, and the recent history of your system.

Installing and Removing the VSM Option

2.1 Introduction

This chapter contains the following information:

- Section 2.2 lists the components of the VSM option and describes the procedures you will use for the initial installation on the system.
- Section 2.3 describes the procedures you will use to deactivate and remove the VSM option so that you can use a different measurement option.

2.2 Initial Installation of the Hardware and Software

This section describes the procedures you will use for the initial installation of the Quantum Design Vibrating Sample Magnetometer (VSM). These procedures apply *only* to the first time you set up and use the VSM option. To re-install the VSM option after it has been deactivated and a different measurement option (e.g., the Heat Capacity option) has been used, you will use the procedures in Section 2.3, "Reconfiguring the system for the VSM Option."

Important: Parts of the initial installation may have been performed at the factory if the VSM option was purchased as part of a new system.

Table 2-1 lists the components of the Quantum Design VSM option. Verify that you have received all the components before you start the installation process.

Table 2-1. VSM system components

COMPONENT	PART NUMBER	ILLUSTRATION
Linear Motor Transport (sometimes referred to as the "Head" or the "VSM Transport")	4096-400	Figures 2-1, 2-6, 5-1
Extender tube flange (sometimes referred to as the "Bottom Weldment Flange") and O-rings	4096-418 or 4096-450 and VON2-030	Figures 2-6, 5-1
Storage Case	4096-150	Figure 5-2
Coilset Assembly**	4096-204 or 4096-600	Figures 2-1, 2-2, 2-3, 5-3–5-4
Sample Tube	4096-301, 4096-620, 4096-350, or 4096-675	Figures 2-1, 2-4, 2-5, 5-5
Sample Rods	4096-352, 4096-610, 4096-275, or 4096-276	Figures 2-1, 5-6
Sample Holders (paddle-shaped)	4096-392	p. 3-4
Sample Holders (trough-shaped)	4096-391	p. 3-5
VSM Powder Capsule pieces	4096-387	p. 3-6
Quartz braces for brass trough	4096-399	
Preamp Cable Assembly	3096-300-01 or 3096-300-02	Figures 2-1, 2-8, 5-9
Motor Drive Cable	3096-200 or 3096-201	Figures 2-1, 2-8, 5-10
Model CM-A Motor Module***	4101-100	Figures 2-1, 2-8, 5-11
Model CM-B VSM Module***	4101-150	Figures 2-1, 2-8, 5-12
Model 1000 Modular Control System****	4100-001	Figures 2-1, 5-13
VSM Option User's Kit	4096-100	Figure 5-7

*This item might be pre-installed. **This item is shipped in the VSM Option User's Kit. ***This item might be pre-installed in the system. ****This item is for the PPMS. VersaLab and Dynacool have system-incorporated module towers.

Installation Process

In the event that you are performing a complete initial installation (i.e., no components were installed at the factory), the process includes the following phases:

- installing and verifying the modular control system, CAN network adapter, and CAN driver software (if no other CAN-based measurement options have been previously installed on your system)
- inserting the control modules
- warming the sample chamber, setting the magnetic field to zero, and venting the sample chamber

- installing the VSM coilset puck
- inserting the VSM sample tube
- mounting the linear motor transport
- completing the electrical connections
- installing the MultiVu software application and the VSM software
- activating the VSM option
- configuring the coilset

The complete initial installation of the VSM option should take no longer than an hour.

In the event that you are performing a partial installation only, check the instructions for each phase to be sure that you understand critical aspects of the process.

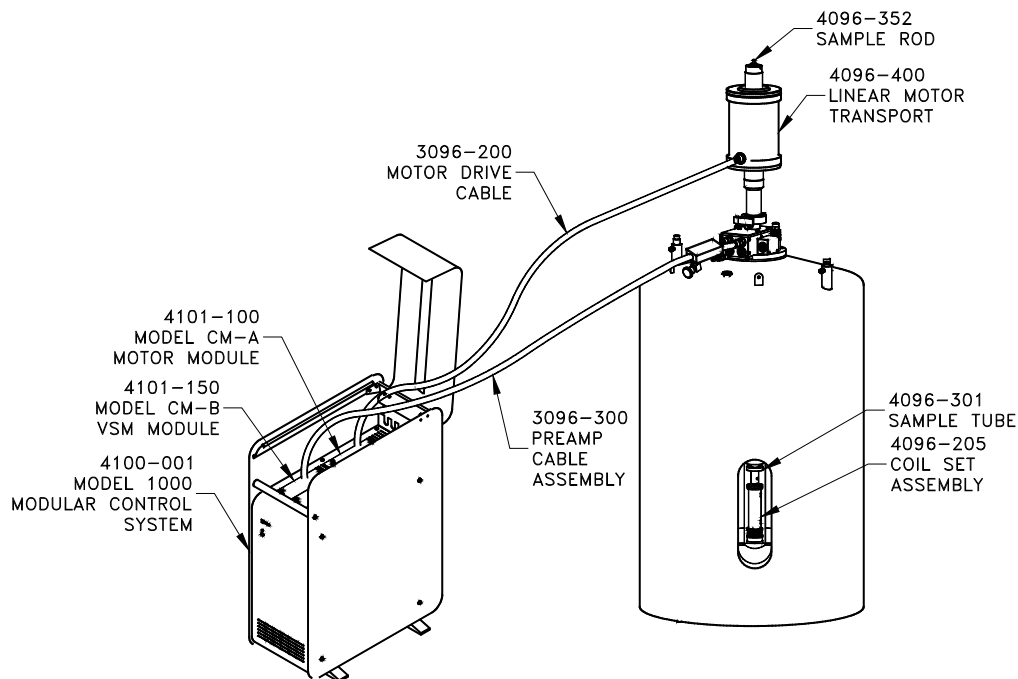


Figure 2-1. System components for PPMS VSM option

Note: The general setup of the VSM option will vary between system platforms.

2.2.1 Install the Modular Control System and CAN Network Adapter (PPMS ONLY)

You will use the instructions in this section only if a Model 1000 modular control system and the driver software have not yet been installed on your PPMS and PC. If your system already has been configured to use Quantum Design modules, you can go to Section 2.2.2.

To install the Model 1000, CAN network adapter, and CAN Manager driver software, refer to the *Model 1000 Modular Control System User's Manual*.

System Verification

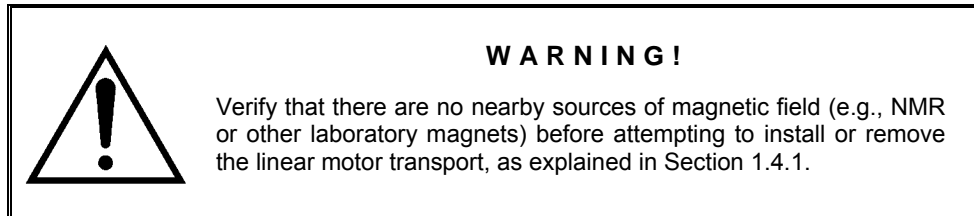
After you have completed installation of the Model 1000 and other components, perform a verification of the system:

1. Verify that the power cable has been connected to the Model 1000 and it has been turned on.
2. Verify that the power LED on the front of the Model 1000 is lit green.
3. Verify that the Model 1000 is connected to the CAN adapter on the PC via the CAN network cable.

2.2.2 Prepare the System for Option Installation

To prepare the System for installation of the VSM option, you will use the MultiVu application to warm the sample chamber to 300K, set the magnetic field to zero (0) Oe, and vent the sample chamber. Then, you will remove any sample puck or option that is currently installed in the chamber. When you do this, be sure to remove the standard centering ring from the chamber opening (the VSM has a custom-designed centering ring).

1. Set the system temperature to 300 K:
2. Set the field to zero (0) Oe.



3. Vent the sample chamber.
4. Remove any sample puck or option that is installed in the sample chamber.
5. Remove the standard centering ring (or any other hardware that is present) from the top flange of the system.

2.2.3 Install the Coilset Puck

The coilset puck contains the VSM detection coils and a thermometer for monitoring the sample temperature. You will insert the coilset puck into the sample chamber by using the standard puck-insertion tool¹ and the same procedures that are used to insert other types of pucks (see the system User Manual for more information). **Install the puck before you insert the sample tube.**

1. On the system, verify that all items have been removed from the sample-chamber opening, including the standard centering ring.
2. Locate the serial number of the detection coilset puck (4096-204 or 4096-600), as shown in Figure 2-2. This serial number will be used to identify the calibration data for the coilset in a later step.
3. Insert the coilset into the sample chamber by using the puck-insertion tool, as illustrated in Figure 2-3.

¹ The puck-insertion tool is also referred to as the puck-extraction tool, the sample-holder tool, and the sample-insertion (sample-extraction) tool, depending on context.

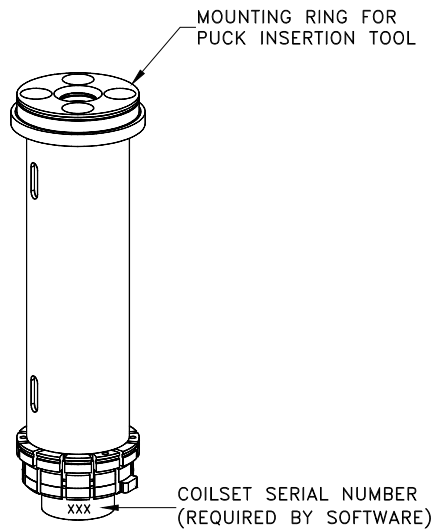


Figure 2-2. Coilset puck (4096-204 or 4096-600), with arrows to coilset serial number and to mounting ring for attaching the puck-insertion tool

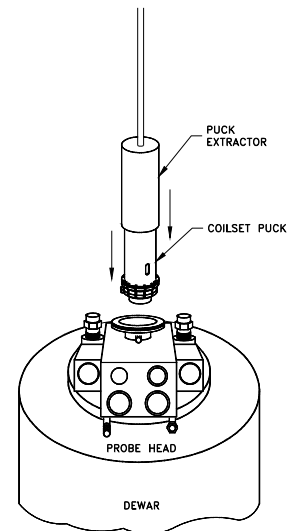


Figure 2-3. Installing the VSM coilset puck with the puck-insertion tool.

2.2.4 Insert the Sample Tube

The sample tube contains low-friction bearing sleeves to center the sample rod in the bore of the coilset. Figure 2-4 shows the sample-tube assembly, where you can see that the top of the sample tube assembly includes an integrated centering ring and a stabilizer post. When the sample tube has been inserted into the sample chamber, the stabilizer post will extend into both the sample chamber and the extender tube flange on the VSM linear motor transport, as is shown in Figure 2-5. The primary functions of the post are to act as a guide when the transport is installed and to keep the transport on the system. See Chapter 5 for more information on the sample tube.

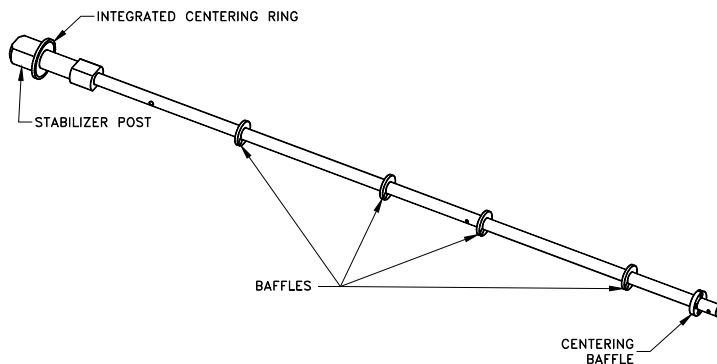


Figure 2-4. VSM sample-tube assembly (4096-301 is shown for example)

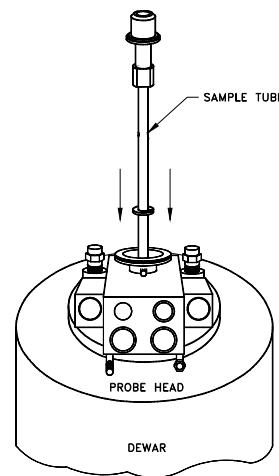


Figure 2-5. Inserting the VSM sample tube into the sample chamber


Use the steps below to install the sample-tube assembly.

1. Verify that the standard centering ring has been removed from the top of the system. (A VSM-specific centering-ring assembly has been integrated into the VSM sample-tube assembly.)

Important: You cannot use a standard centering ring between the VSM linear motor transport and the sample chamber. As a safety mechanism, the VSM system cannot be installed on the system without the VSM-specific components, such as the VSM sample-tube assembly with its integrated centering-ring and stabilizer post.

2. Examine the O-ring on the sample-tube centering ring for dust or dirt. If it is dirty, clean it and lightly grease it with silicone vacuum grease.
3. Using Figure 2-5 as a guide, carefully lower the sample tube assembly into the sample chamber until the VSM centering ring seats onto the top flange.

2.2.5 Mount the VSM Linear Motor Transport



WARNING!

Verify that there are no nearby sources of magnetic field (e.g., NMR or other laboratory magnets) before attempting to install or remove the linear motor transport, as explained in Section 1.4.1.

The VSM linear motor transport (Figure 2-6) moves the sample. You will mount the linear motor transport directly on top of the sample-chamber opening after you have inserted the sample tube into the sample chamber. Before you install the linear motor transport, you must remove the shipping plug and install the extender-tube-flange assembly. See Chapter 5 for more information on the linear motor transport and its operations.

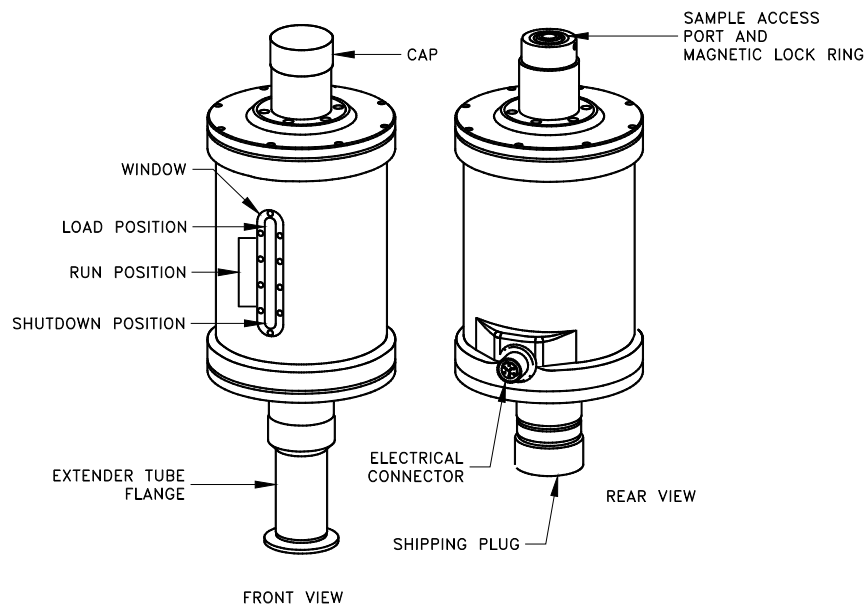


Figure 2-6. Front and rear views of the VSM linear motor transport (4096-400). The rear view (right) shows the transport with the shipping plug installed; in the front view (left), the shipping plug has been replaced by the extender tube flange.

CAUTION!

- ◆ Use care when installing the VSM linear motor transport—it is fragile, bulky, and moderately heavy (about 10 kg or 22 lb).
- ◆ Always use the VSM-specific parts (e.g., the centering rings) to ensure that the equipment operates safely and properly.
- ◆ Always use the flange clamp (Figure 2-7) to hold the linear motor transport onto the stabilizer post.

1. Prepare the extender tube flange:
 - Locate the extender tube flange and the flange O-ring (part number VON2-030).
 - Wipe the neck of the flange with a lint-free cloth (e.g., Kimwipe) to remove any dust or dirt.
 - Place the O-ring into the neck of the extender tube flange. Firmly press on the O-ring to assure it is completely seated.
 - Wipe the O-ring and lightly grease it with silicon vacuum grease.
 - Place the extender tube flange on a clean piece of paper or lint-free cloth until it can be installed.
2. Remove the VSM linear motor transport and its stand from the storage case and place them on a stable work surface, keeping the motor in a vertical position.
3. Remove the shipping plug from the bottom of the motor (see Figure 2-6).
4. Verify that the O-ring of the extender tube flange is still in place and its exposed surface is clean. If it is dusty or dirty, clean it and lightly grease it with silicon vacuum grease.
5. Screw the extender tube flange onto the bottom of the motor until it is tightly attached, using Figure 2-6 for an example. Hand tighten the tube **only**.
6. Remove the VSM linear motor transport from the stand, keeping it upright. For example, you can support the weight of the linear motor transport by gripping it with one hand on the top tube and the other hand on the extender tube flange.
7. Place the VSM linear motor transport onto the top flange of the system by orienting the electrical connector to the rear of the cryostat. The linear motor transport should slide over the stabilizer post at the top of the VSM sample-tube assembly. Figure 2-7 shows where the stabilizer post emerges from the sample chamber, the correct orientation of the linear motor transport, and other relevant parts of the equipment.
8. Verify that the integrated VSM centering ring is sandwiched snugly between the top flange and the linear motor transport.
9. Attach the flange clamp to the flange. **Always use the flange clamp to hold the linear motor transport onto the stabilizer post.**

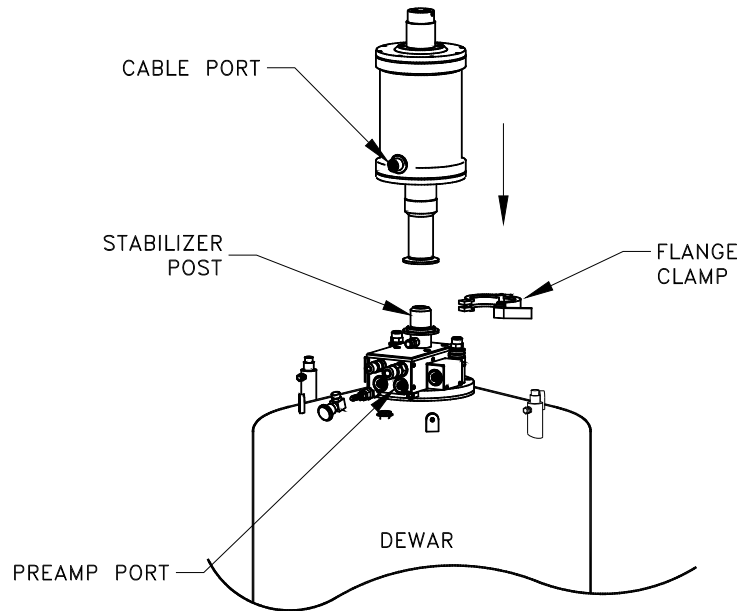


Figure 2-7. Installing the VSM linear motor transport on the top flange

2.2.6 Complete the System Connections

Using Figure 2-8 for guidance, complete the electrical connections for the VSM option. After you have attached the connectors, verify that the connections are firm.

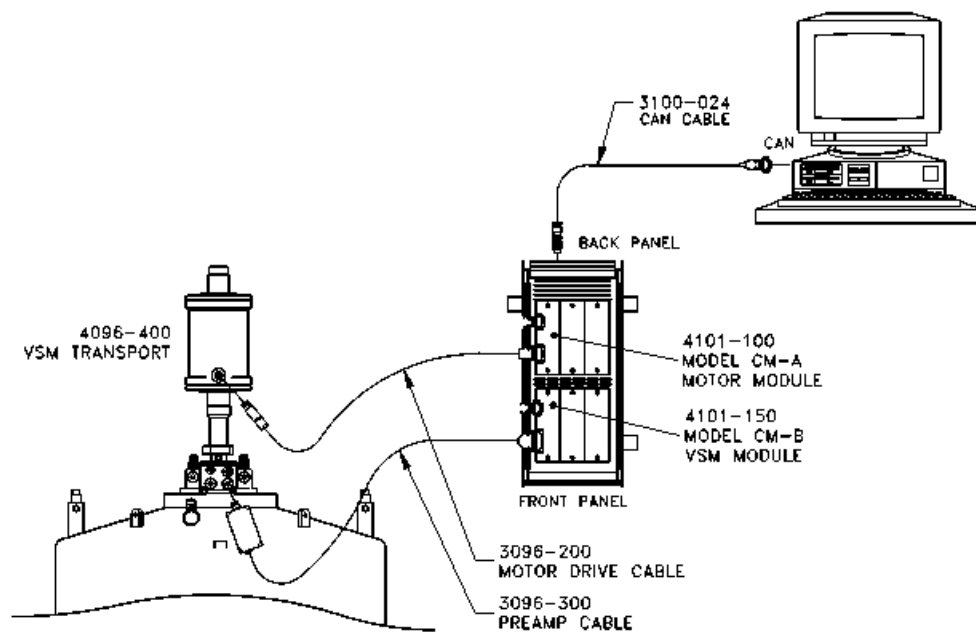


Figure 2-8. VSM option connections on a PPMS

2.2.7 Install the VSM Software

Use the following instructions to install the MultiVu and VSM software applications on your PC. If you purchased the VSM option as part of a new system, you can go to Step 3 below, "Verify that the VSM software is properly installed . . ." See Chapter 6 for more information on the VSM application.

1. Install the most recent version of the MultiVu software if it is not already installed.
2. Install the VSM software by starting the VSM software setup wizard and following the instructions.
3. Verify that the VSM software is properly installed by activating it from within MultiVu:
 - a. Start the MultiVu application program.
 - b. Go to the **Utilities** menu on the main MultiVu menu bar (at the top of the application window).
 - c. Select **Utilities >> Activate Option** (Figure 2-9).

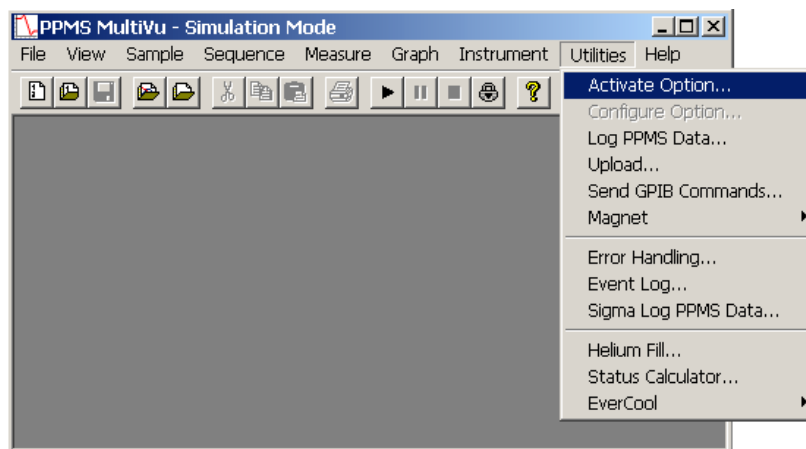


Figure 2-9. PPMS MultiVu menu bar and Utilities dropdown menu with Activate Option selected

4. Three events will occur as soon as you have activated the VSM option.
 - The **VSM Log** window and the **VSM Control Center** will open (see Figure 2-10). In the control center you will see four panels or "tabs": **Install**, **Data File**, **Sample**, and **Advanced**. The **Install** tab is usually at the front when the control center opens. (When running in simulation mode, the **VSM Control Center** is titled **VSM SIM**.) Note the **Configure VSM System** button under the **Chamber Status** area of the **Install** panel; you will use this button to verify and test the coilset calibration in the next phase.
 - The VSM linear motor transport will perform a **Home** (or homing) operation. During a homing operation the system finds the full range of travel for the transport by going through a full travel cycle. The cycle ends at the top in the sample-load position.
 - The **View**, **Sample**, and **Measure** menus on the MultiVu menu bar will show VSM-specific features. For example, the **Status** bar at the bottom of the **VSM Control Center** (Figure 2-10) reads "VSM Ready," and VSM-specific commands are accessible on the **Measure** dropdown menu (Figure 2-11). See Chapter 6 for a full description of the VSM software.

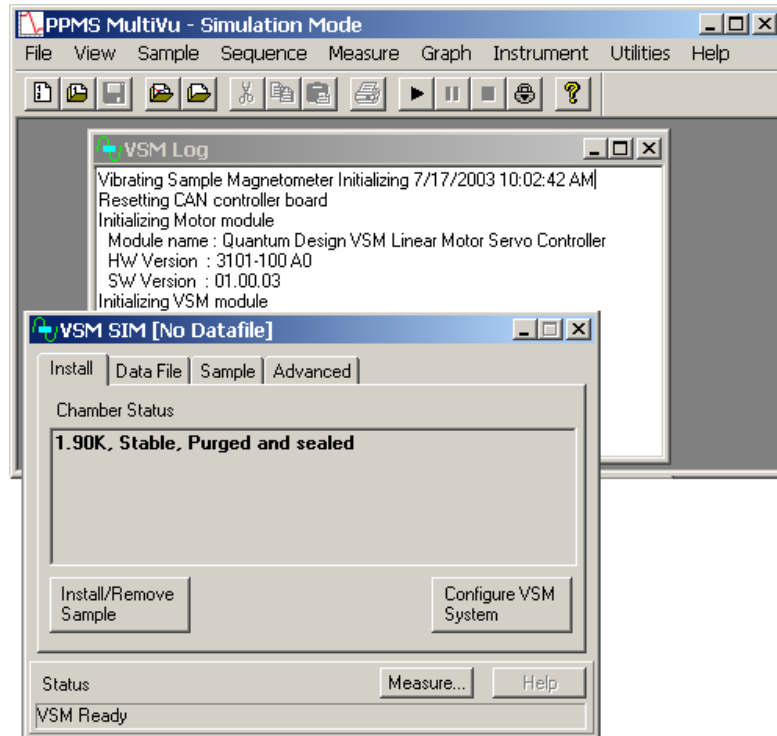


Figure 2-10. VSM Control Center and VSM Log window

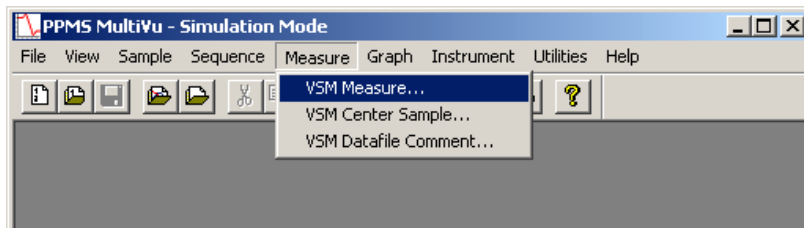


Figure 2-11. PPMS MultiVu menu bar and Measure dropdown menu for VSM option

5. Next, you will verify the serial number on the detection coilset puck and test the coilset thermometer and system calibration.

2.2.8 Configure the VSM System

The serial number of the detection coilset puck identifies the calibration file that is used to calibrate the coilset. You must verify that the serial number on the puck, which you obtained in Section 2.2.4, matches the standard calibration file referenced in the software. You will use the **Configure VSM System** dialogs to specify the puck serial number and test the VSM hardware.

1. Locate the puck serial number that you obtained in Section 2.2.3.
2. Click on the **Configure VSM System** button, which is located on the right-hand side of the **Install** tab (Figure 2-10).
3. Page 1 of the **Configure VSM System** dialog will open (Figure 2-12). Note the text box where you will enter the serial number of the standard VSM calibration file.

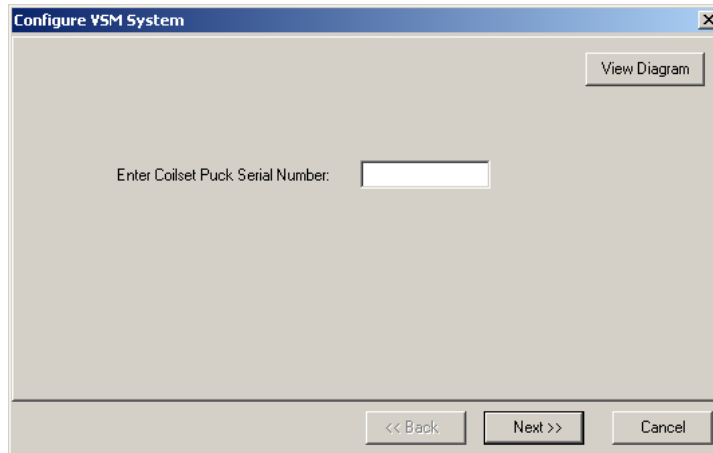


Figure 2-12. Configure VSM System dialog, page 1

4. Verify that the number in the text box matches the serial number of your detection coilset puck. If no number is displayed (as above), enter the serial number of your detection coilset puck in the text box.
5. Click on the **Next >>** button at the bottom of the dialog box. Page 2 of the **Configure VSM System** dialog (Figure 2-13) will open, showing the results of system tests to verify system operations.

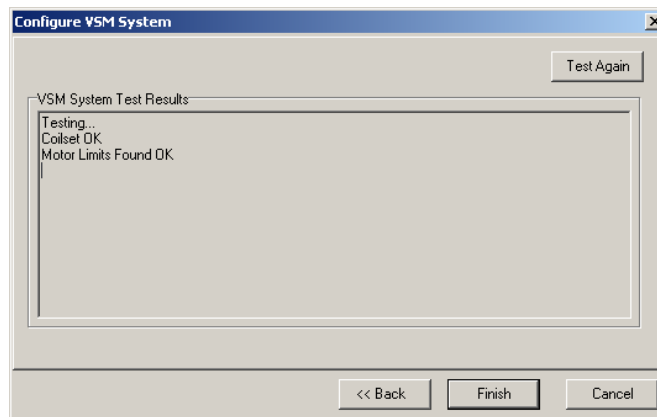


Figure 2-13. Configure VSM System dialog, page 2: Testing calibration file

6. When the report in the **VSM System Test Results** area is complete and no errors are reported, click on the **Finish** button. The **Configure VSM System** dialog will close and you will be back at the **VSM Control Center** and the **Install** tab.
7. If you have completely followed the VSM hardware and software setup steps, the system is now ready for you to mount a sample, install it in the cryostat, and perform VSM measurements, as is explained in Chapters 3 and 4. First, please review Section 1.4, "Safety Precautions," for important information.

2.3 Removing the VSM Option



WARNING!

Verify that there are no nearby sources of magnetic field (e.g., NMR or other laboratory magnets) before attempting to install or remove the linear motor transport, as explained in Section 1.4.1.

You do not need to remove the VSM linear motor transport and its associated hardware from the system while it is idle. However, if you intend to use it for other types of measurements (e.g., Heat Capacity, Thermal Transport), you must first remove the VSM option.

As summarized below, you will use the **VSM Install/Remove Sample Wizard** to prepare the system so that you can remove the linear motor transport, sample tube, and coilset puck. These procedures are essentially the reverse of the installation procedure.

Summary of VSM Removal Procedures

1. Prepare for removal of the VSM transport and hardware:
 - a. Set the field to zero.
 - b. Use the **VSM Install/Remove Sample Wizard** to warm the sample chamber to 300 K, vent the chamber, and move the transport to the load position.
 - c. Remove the sample rod.
 - d. Shut down the linear motor transport.
2. Deactivate the VSM software application.
3. Remove the linear motor transport and place it in the storage case.
4. Remove the sample tube and the coilset puck.

2.3.1 Prepare for Removal

1. If necessary, activate the VSM software from MultiVu (select **Utilities >> Activate Option >> VSM**) as explained in Section 2.2.7.
 - If the field is not zero, set it to
 - Use the **VSM Install/Remove Sample Wizard** to remove the sample rod.

CAUTION!

Verify that you have removed the sample rod before continuing.

2. To continue with the VSM removal process, end the **VSM Install/Remove Sample Wizard** by clicking on the **Cancel** button. This button will close the install dialog and return you to the **VSM Control Center** and the **Install** tab. Before you can remove the linear motor transport, you must deactivate the VSM option, as is explained in Section 2.3.2.

2.3.2 Deactivate the VSM Option

1. Select **Utilities >> Activate Option** from the dropdown menu of the MultiVu window (Figure 2-9).
2. When the **Option Manager** dialog opens, select **VSM** and click on the **Deactivate** button. This will move the VSM option from the **Active Options** section of the dialog to the **Available Options** section. The **VSM Control Center** and the **VSM Log** window will close, but the MultiVu software application will remain open.
3. Continue with the VSM removal procedures below.

2.3.3 Remove the VSM Linear Motor Transport

1. Unplug the electrical connector from the back of the VSM linear motor transport. (You can leave the other end of the cable connected to the Motor Module.)
Important: Never attempt to move the linear motor transport when it has a cable connected to it.
2. Remove the flange clamp from the top flange of the sample chamber (see Figure 2-7).
3. Slowly lift the linear motor transport until it has cleared the stabilizer post (see Figure 2-7).
4. Place the linear motor transport back in the storage case (4096-150).



WARNING!

Store the VSM linear motor transport in a secure location to prevent it from being attracted to magnetic fields in the laboratory, including those produced by the superconducting magnet, as explained in Section 1.4.1.

2.3.4 Remove the VSM Sample Tube and Coilset Puck

1. Remove the VSM sample tube from the sample chamber.
2. Remove the VSM coilset puck from the sample chamber by using the puck-extraction tool.²
3. Unplug the VSM preamp cable from the probe head and set it aside. You do not need to disconnect the other end of the cable from the Motor Module.
4. Return the blank flange to the top of the probe head or install another of the Quantum Design measurement options.
5. When the sample chamber has been closed, you can purge and seal it by using the **Chamber** dialog box.
 - Select **Instrument >> Chamber**.
 - In the **Chamber** dialog box, click on the **Purge/Seal** button.
6. The base measurement system is now ready for you to install a different option.

² See Footnote 1.

Sample Preparation and Mounting

3.1 Introduction

This chapter contains the following information:

- Section 3.2 discusses constraints on the samples that can be measured with the VSM option.
- Section 3.3 explains how to mount samples for measurement with the VSM option.

3.2 Sample Properties

The quality of your VSM measurement results will be affected by the dimensions and shape of the sample and the size of its magnetic moment.

3.2.1 Size and Shape

The geometry of the detection coils in the VSM constrains the dimensions of samples that can be measured. Figure 5-4 gives the dimensions of a standard coilset puck. In order for the sample and sample holder to fit into the detection coils without a high risk of rubbing against the coil set bore, their diameter should be less than 4 mm. Frictional heating (especially at low temperatures) and noise in the VSM measurements at high fields are common symptoms of friction between the sample holder and coil set. Furthermore, accurate results require samples that have a small vertical size compared to the baseline dimension of the detection coils. For the standard coilset puck, this baseline is 7.11 mm. In practice, a cylindrical sample in the shape of the included palladium standard sample is close to ideal.

Table 3-1 shows the calculated effect of the sample length and diameter on the reported sample moment. The entries in the table were calculated for a vertically oriented cylindrical sample of length L and diameter D . Each entry corresponds to the ratio of the reported moment to the true moment. For example, for a sample of length $L = 5.0$ mm and diameter $D = 2.0$ mm measured with a peak amplitude $A = 2.0$ mm, the software will report a numerical result for the moment that is 0.9476 times the answer obtained for a pointlike sample of the same total moment. Note that while the values in Table 3-1 have been normalized to 1.0000 for a pointlike sample, the VSM system is calibrated against the palladium standard sample ($L=3.8$ mm, $D=2.8$ mm) at a peak amplitude $A = 1.0$ mm.

Table 3-1. Calculated ratio of the reported moment to the true moment for different size cylindrical samples and different amplitudes.

SAMPLE DIMENSIONS		PEAK AMPLITUDE OF SAMPLE OSCILLATION		
Length <i>L</i> (mm)	Diameter <i>D</i> (mm)	0.5 mm	1.0 mm	2.0 mm
0	0	1.0000	1.0000	1.0000
0	1	1.0003	1.0005	1.0014
0	2	1.0007	1.0017	1.0053
0	3	1.0005	1.0026	1.0109
1	0	0.9996	0.9993	0.9981
1	1	0.9999	0.9999	0.9996
1	2	1.0007	1.0013	1.0037
1	3	1.0010	1.0028	1.0097
2	0	0.9978	0.9966	0.9921
2	1	0.9985	0.9975	0.9937
2	2	1.0002	0.9999	0.9986
2	3	1.0020	1.0029	1.0059
3	0	0.9933	0.9906	0.9808
3	1	0.9944	0.9920	0.9828
3	2	0.9976	0.9958	0.9887
3	3	1.0020	1.0013	0.9980
5	0	0.9662	0.9597	0.9367
5	1	0.9687	0.9622	0.9394
5	2	0.9759	0.9698	0.9476
5	3	0.9877	0.9820	0.9613
10	0	0.6961	0.6889	0.6647
10	1	0.6988	0.6914	0.6669
10	2	0.7070	0.6993	0.6737
10	3	0.7213	0.7130	0.6854

Table 3-1 only addresses pointlike and cylindrical samples, but you can obtain similar results when you measure samples with other shapes. In such cases, use Table 3-1 as a rough guide to approximate the expected errors. Again, detection-coil sensitivity for the included palladium VSM standard sample (4096-390) is not necessarily the same as detection-coil sensitivity for samples with shapes and/or sizes that differ from the included palladium standard sample. The VSM measurement software will not correct for such differences.

3.2.2 Errors from Radial Offset of Sample

You also should consider the radial centering of the sample in the coilset when the absolute accuracy of VSM measurement results is important.

Table 3-2 shows the calculated effect of radial offset for cylindrical samples of different sizes, using oscillation amplitudes of 1 mm and 2 mm. Note that when the centering error is only 1 mm there could be as much as 1% error in the repeated moment.

Table 3-2. Calculated ratio of the reported moment to the true moment of cylindrical samples for different radial offsets from the coilset centerline.

PEAK AMPLITUDE	SAMPLE DIMENSIONS		SAMPLE RADIAL OFFSET				
	A (mm)	L (mm)	D (mm)	0 mm	0.5 mm	1.0 mm	1.5 mm
1.0	1.0	1.0	1.000	1.001	1.003	1.003	0.997
1.0	2.0	2.0	1.000	1.001	1.003	1.005	1.000
1.0	3.0	3.0	1.001	1.003	1.006	1.009	1.006
2.0	1.0	1.0	1.000	1.002	1.010	1.020	1.028
2.0	2.0	2.0	0.999	1.002	1.010	1.020	1.030
2.0	3.0	3.0	0.998	1.001	1.010	1.023	1.035

3.2.3 Size of Magnetic Moment

The magnetic moment of your samples should be larger than about 10^{-6} emu to be detected in the VSM.

3.3 Mounting Samples

3.3.1 Accurate Sample Location

The VSM system uses a touchdown technique for automatic centering of the sample in the detection coils. This technique is described in detail in Chapter 4. To optimize the touchdown, take special care with two steps in your preparations:

- Verify that the sample is mounted on the sample holder near 35 mm from the bottom of the sample holder. Quantum Design has provided you with a special sample-mounting fixture (see Figures 4-2 and 5-8) for locating the sample.
- Verify that the end of the sample holder has a very well defined contact surface for performing the touchdown operation.

Quantum Design recommends that you locate the sample at an offset of 35 mm. The height of the coilset is about 40 mm, so the offset of 35 mm places the end of the sample holder 5 mm above the puck surface. This location is far enough from the end of the sample holder so that end effects are minimal, yet it safely allows up to 4 mm oscillation amplitude of the sample rod. In addition, use of this location exposes the sample to minimal variations in applied magnetic field, because the sample is only moved 5 mm during a touchdown operation.

3.3.2 VSM Sample Mounting Techniques (Application Note 1096-306)

The techniques being illustrated will apply to the SQUID-VSM, PPMS-VSM and VersaLab cryogen-free VSM systems. Quantum Design provides the selected sample holders. The materials for securing the sample to the holders are not included with the installation. Due to high accelerations in VSM measurements, secure sample mounting is more challenging than in most other measurements. Loosely mounted samples will result in noise in the moment measurement, see app note 1096-303 and 1500-009 on the Quantum Design website. Axial and radial sample positioning error introduces inaccuracy of the reported moment, see app note 1500-010. Also, a sample geometry influences accuracy of the reported moment, see app note 1500-015. Keep the sample length less than 5 mm to maintain the accuracy of the point source dipole approximation.

1. The **sample holders supplied by Quantum Design** are designed to fit on the mounting station. The total length of the holder and exact location of the sample are better defined by using the mounting station. While the fragile quartz paddle is designed for the lowest moment samples, a more robust brass trough provides versatility.
 - A. The **quartz paddle** is redrawn from a 4 mm diameter rod. Elimination of surface microcracks improves mechanical strength of the quartz paddle. A nitric acid dip removes surface impurities. Smooth surface can make cleaning easy but reduces adhesion of glue. The adapter is made of glass-filled polycarbonate, which should never be exposed to organic solvents, especially acetone. The outer gluing surfaces of the quartz pieces are sanded to help bind with the epoxy. A complimentary shaped shim is added to the paddle and put inside the adapter containing epoxy resin. The epoxy is specially chosen to withstand both high temperatures (400 K) and cryogenic conditions. The assembly is cured straight in a custom fixture.



1. For mounting **thin films parallel to field** (see figure to the left), do not exceed the width of the paddle. Wider samples can be mechanically secured and better protected using the brass trough or aluminum frame techniques. Place the quartz holder in mounting station. Using a sharp wooden stick or other tool, place a small drop of glue on the paddle, set film on top and press to secure bond. Let it dry completely at ambient conditions. If the sample can handle the heat, increase temperature to 340 K and purge sample chamber. Return to 300 K, purge and seal, or lower temperature and follow centering routine. To remove sample, hold length of quartz paddle with no pressure on adapter junction. A thin wooden flat can transport solvent under the chip. Wait for solvent to penetrate. Free the sample with appropriate leverage.
2. For **single crystal** samples and low temperature analysis, put GE 7031 on crystal then secure to quartz. A small amount of **fine powder** can be mixed into a drop of varnish, although it is difficult to get a quantitative mass with this technique. If taken to 340 K to cure before completely dry, a large field like 5 Tesla should result in the alignment along a preferred crystallographic axis which is frozen in place by going cold. Kapton tape is also effective way to secure small amounts of material, but keep sample as point source and tape symmetrical along the axis of motion. Concern with tape is the random contamination from dust in a lab environment.
3. To **clean the quartz paddle** use a solvent specific to the glue. Cotton swabs help keep all solvents away from the polycarbonate adapter, especially acetone. Do not use sonicator, which could introduce cracks at the junction points. The common break point is at the adapter junction and caused by lateral force on the sample rod. Frequently, breakage occurs during the sample mounting and cleaning process.

- B. The **brass trough** is made from cartridge brass tubing with a cobalt-hardened gold plating finish. The adapter is made of glass filled polycarbonate, which should never be exposed to organic solvents, especially acetone. After the assembly is fit together, the added epoxy is allowed to cure straight in a fixture. In the case of PPMS and VersaLab platforms, brass trough holders come in a small or large diameter which fit samples of diameter in the range of 4 or 6mm, respectively.



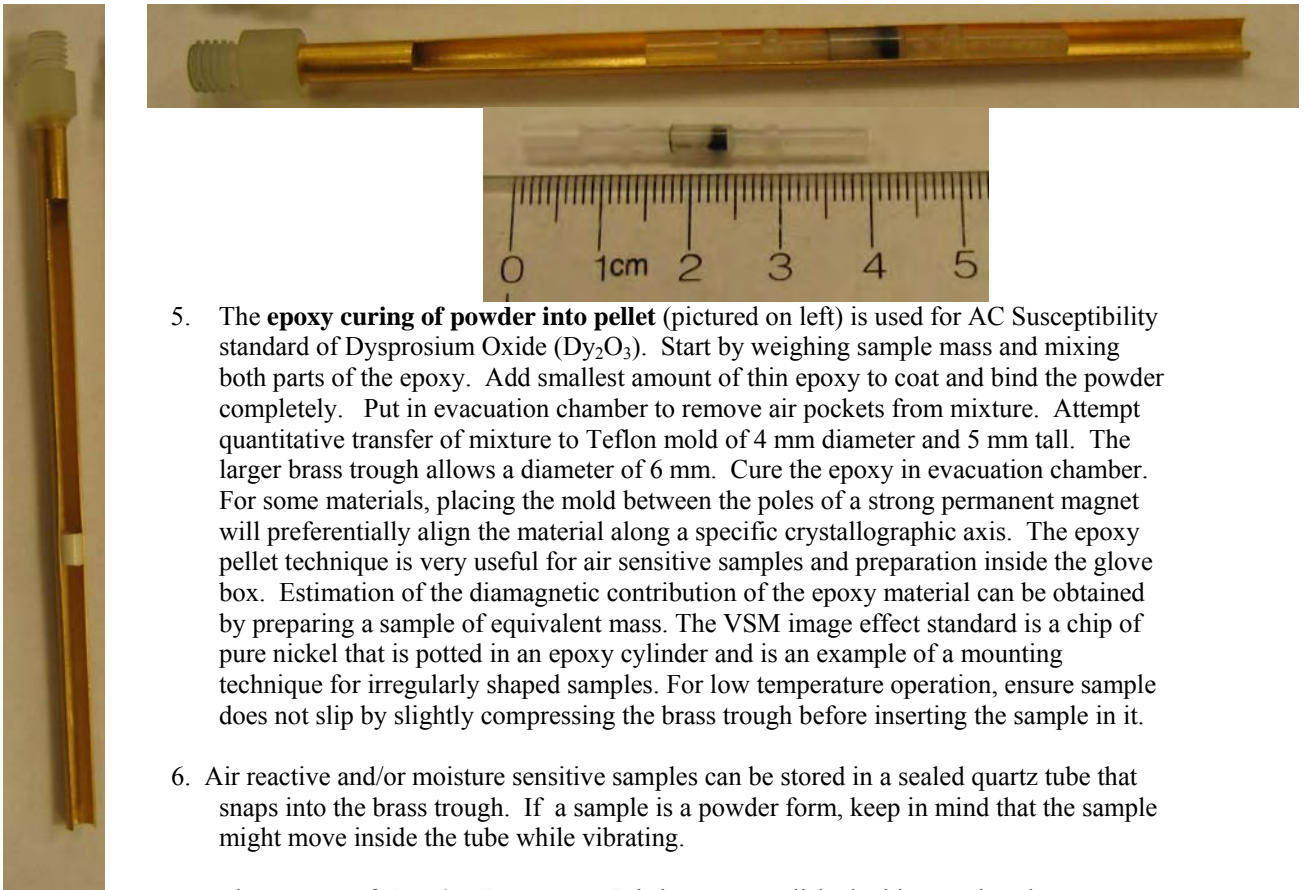
1. The **palladium standard** (pictured) is mounted with GE-7031 varnish to help withstand thermal cycles during installation tests. The only function is calibration of magnetometer at 1 Tesla and 298 K. To get the expected moment, simply multiply the mass of palladium, the applied field and the susceptibility, which is 5.25×10^{-2} emu/gram-Tesla at 298 K. The expected moment at 1 Tesla for a 0.25 gram cylindrical shape is 0.013 emu. While magnetization versus temperature for palladium has characteristic features, it is not a suitable thermal reference standard. Consider temperature independent diamagnetic Quantalloy or the Curie-Weiss paramagnet dysprosium oxide pellet. Even at the lowest temperatures, the contribution from impurities in the varnish is below 0.1% of the large Pd moment.
2. For **films parallel to field**, the best technique for lowest background is to simply press fit with tension so the film acts as the point source dipole. Contamination of the inner walls of the trough is more likely through scratches. Adding glue or varnish should prevent the sample from becoming loose. A long (10 cm) Quantalloy support piece can be wedged into place and the films attached to this surface with glue. It is more easily cleaned, disposable and will not magnetically contaminate the brass trough. The short (4 mm tall) Quantalloy coupon is useful for sensitive oxygen leak tests near 50 K by providing a large surface area, point source dipole and diamagnetic signal at 1 Tesla. After the quartz sample holder breaks, the brass trough can be made to accommodate the inverted shape. The quartz provides a magnetically clean surface area.
3. For **films perpendicular to field**, an effective technique uses quartz braces that snap into the brass trough. The length of the brace will depend on the system, with the SQUID VSM needing the longest to minimize end effect issues. The braces must come into contact with both sides of the film to hold it securely. If contact with the surface is not allowed, glue sample to one end and put second quartz brace as close as possible to minimize gap signal. Matched sets of fixed angle quartz pieces with values such as 22.5, 30, 45 and 60 degrees can provide a useful data set for verification of rotator experiments. While the press fit of metal on quartz should be good over the range of 1.8 K to 400 K, it is prudent to secure ends of quartz pieces with glue. If the sample signal is comparable or smaller than the brass trough, one should use a quartz paddle. If the film is deposited on the silicon substrate (100), it is easy to cut a rectangular or square piece along (110) directions. Place two pieces of samples with a film side face to face and mount the pair vertically on the quartz paddle with an appropriate fixture using varnish or one of the glues. Two pieces of films increase the signal and help them stand perpendicular to the quartz paddle.
4. **Polypropylene powder holder** is an injection molded plastic part that snaps into the brass trough. The figure below shows the shapes of all are identical. Sample mass from 1 mg to 10 mg of powder is put into the opening. A second piece will compress the powder and seal the combination. The assembly snaps into the brass trough for a secure press fit. The material covers the entire temperature range from 1.8 K to 400 K. This powder holder is not recommended for liquid samples, so please refer to the section on custom mounting techniques.

A guide to the complete experiment is as follows:

- Inspect and clean the brass trough and both polypropylene powder holders.

- Allow for the appropriate sized gap as the 2 pieces are joined as will occur when the sample is later used. Snap rigidly into place within the brass trough. Measure the distance from end of brass trough to center of gap location using mounting station.
- Insert sample holder and aligned rod into VSM at 300 K and nearest zero field. Use wizard to manually locate the sample position as previously measured.
- Run exact sequence to be performed when the sample is present. The sensitivity and precision of the VSM style lock-in technique allows accurate background determination, even though a center response function may not be obtainable.
- After sequence complete, remove parts from brass trough. Weigh both pieces, put powder inside the opening of one and securely close with the remaining piece. Weigh mass of assembly in addition to keeping track of mass change in source.
- Secure in brass trough and accurately measure location of gap using mounting station.
- At first, use wizard to manually locate sample position. Once field is applied, perform centering function. Repeat sequence run with the blank holder.
- The use of standard materials of similar dimensions and signal should be run for verification of the accuracy of the process. We have shown that remarkably sensitive measurements are easily obtained, especially for low field magnetization versus temperature analysis.

A video of this process is available from Quantum Design. Please e-mail request to applications@qdusa.com



5. The **epoxy curing of powder into pellet** (pictured on left) is used for AC Susceptibility standard of Dysprosium Oxide (Dy_2O_3). Start by weighing sample mass and mixing both parts of the epoxy. Add smallest amount of thin epoxy to coat and bind the powder completely. Put in evacuation chamber to remove air pockets from mixture. Attempt quantitative transfer of mixture to Teflon mold of 4 mm diameter and 5 mm tall. The larger brass trough allows a diameter of 6 mm. Cure the epoxy in evacuation chamber. For some materials, placing the mold between the poles of a strong permanent magnet will preferentially align the material along a specific crystallographic axis. The epoxy pellet technique is very useful for air sensitive samples and preparation inside the glove box. Estimation of the diamagnetic contribution of the epoxy material can be obtained by preparing a sample of equivalent mass. The VSM image effect standard is a chip of pure nickel that is potted in an epoxy cylinder and is an example of a mounting technique for irregularly shaped samples. For low temperature operation, ensure sample does not slip by slightly compressing the brass trough before inserting the sample in it.
6. Air reactive and/or moisture sensitive samples can be stored in a sealed quartz tube that snaps into the brass trough. If a sample is a powder form, keep in mind that the sample might move inside the tube while vibrating.
7. The process of **cleaning brass trough** is best accomplished with organic solvent suitable for the dry glue. Even though the brass is gold plated, strong acidic washes should be avoided in case of scratches. While the junction is partly a press fit, the use of

epoxy requires avoiding contact with cleaning solvents. Cotton swab soaked in appropriate solvent and let set against old glue until swab is dry. Apply a new soaked cotton swab to loosened glue and remove without risking the gold plating. Under the general principle of uniform mass of the holder for +/- 3 cm from the sample location, keeping a uniform gold coating is relevant to the background contribution.

Pictures of cleaning supplies:

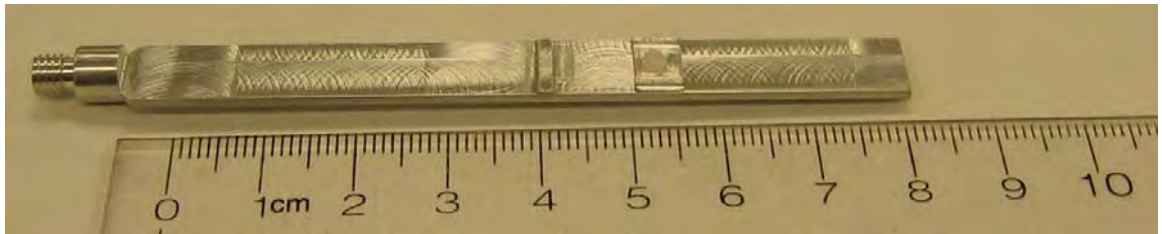
Specific cotton swab for brass trough iso-propanol (IPA), acetone, toluene, nitro-methane



2. Just a few of the **methods and materials for securing the sample** to the holder are presented. The primary goal is to keep the sample a point source dipole during the measurement. Since any glue or tape will introduce point source dipole contributions to the total magnetic moment, the material should be applied sparingly and symmetrically.
 - A. The **press fit** into brass trough holder achieves all the ideal aspects of a secure sample mounting without the need for extra glue. One concern is a transfer of material to the holder, but routine blank measurement checks will confirm the low-level background contribution. The technique is appropriate for samples directly in contact with the brass or through various support mechanisms.
 - B. The term **glue** is loosely applied to any material that will secure a sample to a support material. Generally the materials will harden over time, with the process aided by increasing temperature and keeping under vacuum. Pockets of trapped oxygen or adsorption to very high surface area materials will produce a magnetic signature around 50 K. Each material has a specific range of conditions for optimal usage; with varnish best at the lowest temperatures and Duco cement the most easily removed. Since the contents of each glue are not normally disclosed by manufacturers, it is hard to know magnetic properties. A general rule is that colorless materials are less magnetic than colored ones because dyes used in the materials tend to have magnetic signature. However, it is best for users to verify the magnetic property of the glue.
 1. The legendary **GE7031 varnish** is a vinyl phenolic adhesive (safety labeling H=1; F=4; R=3; PE=3). As the bulk material in container ages, it will become thicker. Adding iso-propanol (IPA) and/or toluene will thin solution. Cleaning is best aided with toluene. The aging and/or oxidation process will lead to a darker coloring of the solution. This tends to correlate with increased magnetic signature. The ideal solution is a light tan honey colored solution, freely flowing without addition of extra solvents.
 2. Easily obtained **Duco cement** is useful at room temperature and for securing materials of similar thermal properties, like quartz on quartz. Differing materials will force cracking in the brittle Duco cement upon cooling below 150 K. Duco may go on thick but it will dry or cure to a thin residue. A key reason for regular use is Duco solubility in acetone. Powders secured with Duco can be easily recovered by soaking in acetone.

3. Easily obtained **Superglue** (cyanoacrylate) is a fast drying, very secure bonding method for room temperature measurements. The very thin nature allows low mass, low magnetic signature application. At the colder temperatures, it may not hold materials with different thermal properties. The solvent of choice is nitro-methane. Still readily available as a stock car fuel, it should be handled carefully. A cotton swab of nitro-methane with a thin stick to wedge the sample off the holder should work effectively. Please see MSDS for proper ventilation requirements when using these solvents.
 4. The **Zircar cement** is conveniently water based and supplied with the VSM-oven user kit. The primary purpose of application to over 1100 K has been verified. Used in conjunction with tight wrapping of copper foil provides a sufficiently robust design for VSM style measurements. The VSM oven sample holder is not specifically made for low temperature operation. While tests show no harm in thermal cycling to 2 K, there is always a risk of exposing a weakness in the wiring of the heater stick through this procedure. The mounting technique on a blank stick would hold to the lowest temperature. This Zircar cement is also a way to avoid using organic solvent-based materials. The water-based gradual drying process may be appropriate for incorporating powders from biological or other exotic systems. Removal of the sample usually involves a thinly wedged stick providing lateral force on the sample. The cement cracks easily. While water or isopropanol (IPA) may aid in the removal of the excess, the main goal is to not hurt the laminate coating the patterned heater wires. No sharp metal objects, try disposable Teflon tweezers or thin flat wooden surfaces. Use a microscope to see detail of interface.
- C. Tape is a simple and effective method for securing to the quartz rod. It will prevent the sample from being lost during the measurement. The background can be reasonably small if the mass is symmetrical about the sample. Keeping the tape clean from magnetic dust around the lab is the primary concern.
1. **Kapton tape** is commonly used at low temperatures and is available from Quantum Design. It retains properties over entire range of 1.8 K to 400 K. As a point source may show 10^{-6} emu ferro-magnetic signature at room temperature. Like with a straw, if the tape is evenly distributed in the background, there will be no contribution to the reported moment of the sample. To replace common drinking straws, Kapton tubing is available through the medical community and may provide flexibility in diameter selection with lowest level of impurities. Organic solvents like toluene or maybe iso-propanol (IPA) can help dissolve the glue remaining on the holder after tape is removed.
 2. Thin Teflon tape can be wrapped around the quartz holder to secure the sample location without use of glue. However, the technique tends to produce a significant paramagnetic oxygen peak around 50 K. As a precaution, add a heat and purge step to the sequence. The Teflon tape can cover the full temperature range of the instrument, but 350 K is usually sufficient for this purpose. When Teflon becomes a point source dipole like when wrapping the sample, a more significant ferro-magnetic contribution is measured. Each batch should be tested for impurities.
 3. The standard Parafilm roll and various wax materials can be used to secure the sample. Watch maximum exposure temperatures of the wax. These types of materials are typically low moment background and useful with air-sensitive samples or when recoverability is desired.
3. **Custom** mounting techniques for classes of samples:
Note that following techniques may not apply to all platforms.

- A. The hydrostatic **pressure cells** by various vendors require securing the sample for the VSM style measurements. The GE7031 varnish has proven effective for room temperature analysis of dysprosium oxide pellet pieces.
- B. The **Teflon bucket** for organic solvents or air sensitive samples was originally designed for the PPMS-ACMS instrument. The length was shortened for the MPMS XL with RSO instrument and is particularly useful with SQUID based AC susceptibility measurements. Now, there are new lengths for both the PPMS VSM with the large bore coil-set or the SQUID VSM. Simply reverse the blue adapter male threaded piece and glue carbon fiber rod with lid of bucket. The torque and vibrations should require extra security in securing the threaded top. Teflon tape or layer of epoxy on the threads will help maintain a tight fit.
- C. Components like **quartz braces** and Quantalloy supports need to be longer for the SQUID-VSM as compared to the PPMS VSM due to the larger axial dimension of the second order gradiometer.
- D. The **aluminum large area** (pictured below) thin film holder using mechanical technique is only applicable to the large bore coil-set dimensions. The lightweight design allows a large surface area for securing a sample with a clamp and screw mechanical device. It was designed originally for use with 9 x 9 x 0.5mm thin film samples.



- E. **Straws and gelatin capsules are no longer being used.** The commercially available straws are not straight enough to be used for the VSM measurement. The straws tend to bend and make contact with the chamber walls. The frictional heating will restrict the base operational temperature. In addition, the friction of the straw end would cause noise in the VSM measurements. Any loosening of the contact between straw and adapter would result in loss of accuracy that may not be obvious in data set. It is difficult to secure the sample in the straw. Because of these reasons straws are not recommended to use with VSM measurements. Note that SVSM quartz paddle adapters are designed to fit a straw for protecting quartz.
- F. The **quartz tube** is useful to mount a cylindrical sample as well as a bulk sample when the low background moment is important.
- G. **Clear plastic materials** such as polycarbonate, polypropylene, and polystyrene are good candidates for low background moment holders due to their nonmagnetic material characteristics. Avoid using colored plastic materials, since the dyes used to color the plastics are generally magnetic. Keep in mind that even though these plastic materials are good low moment candidates, manufacturing environment might introduce magnetic contaminants to these materials. Users should test these materials for the level of magnetic background.

Please e-mail requests and suggestions to applications@qdusa.com

Part numbers for relevant QD supplies

- 4096-385: Ni image effect assembly (potted)
- 4096-388: Powder capsules, bag of 50 pairs (100 pieces)
- 4096-390: Pd standard assembly in brass trough
- 4096-391: Brass trough sample holder
- 4096-392: Quartz paddle sample holder
- 4096-399: Quartz rod sample brace for brass trough

- 4096-630: LBC brass trough
- 4096-635: LBC aluminum paddle
- 4999-903: Dy2O3 assembly in brass trough
- 4096-030: GE-7031 varnish
- 4097-035: Zircar AL-CEM alumina cement
- ACA 6: Duco cement
- ATK1-025: Kapton tape, 0.001" thick x 0.25" wide

Please contact your local Quantum Design representative for sales inquiries.



Quantum Design supplied sample holders and PPMS-VSM mounting station.



Securing agents from the left: 2-part epoxy; Duco cement; ZircarCement; GE7031 Varnish; fast curing Superglue; slower curing Superglue; Kapton tape.

Taking VSM Measurements

4.1 Introduction

This chapter contains the following information:

- Section 4.2 summarizes the measurement process when you use the Quantum Design VSM option.
- Section 4.3 describes how to install samples for taking VSM measurements.
- Section 4.4 describes how to take immediate-mode measurements with the VSM option.

4.2 Overview of VSM Measurements

A VSM measurement consists of a sequence of centering operations followed by the oscillation of the sample and synchronous detection of the voltage induced in the coilset by the magnetized sample.

In preparation for the measurement, you will use the automated **VSM Install/Remove Sample Wizard** to install the sample. After that, the VSM application software completely automates the next steps, which include centering the sample, setting measurement parameters, and setting up measurement command files as well as operations that are more advanced.

The measurement process and use of the software are explained in the following subsections.

Important: Before you attempt to begin the measurement process, verify that the VSM option and the application software have been installed (Chapter 2) and the sample has been mounted on the sample holder (Chapter 3).

4.2.1 Sample Centering

Accurate VSM measurements require that the system oscillate the sample in the center of the coil pair (see Figure 5-3), and that the centering position be kept stable to within about 0.1 mm at the center of the pickup coils. The 0.1 mm stability is maintained by periodic “touchdown” operations that adjust for changes in the sample position.

4.2.1.1 THE TOUCHDOWN OPERATION

Automatic sample centering is accomplished with the touchdown operation. To perform a touchdown operation, the linear motor transport lowers the sample rod until the end of the sample holder touches the puck surface (see Figure 4-1). At this point, the software knows the precise offset between the coilset and the sample, based on the dimensions of the coilset and the location of the sample on the sample holder. The linear motor transport then moves the center of the sample to the center of the coilset to continue measuring according to the following relation:

$$\text{Measure location} = (\text{touchdown location}) + (\text{coilset height}) - (\text{sample offset}),$$

where coilset height ("H" in Figure 5-4) is a calibrated constant for a given puck and is near 40mm.

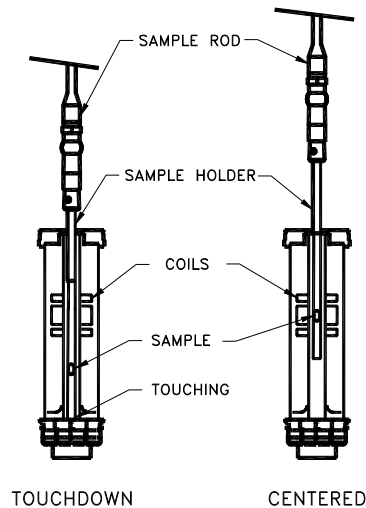


Figure 4-1. The touchdown centering operation

4.2.1.2 SCHEDULING TOUCHDOWN OPERATIONS

The software usually performs touchdown operations automatically at prescribed intervals of temperature (typically 10 K) or time (typically 10 minutes). Touchdowns can also be performed explicitly in a sequence (**VSM >> Center Sample**) in immediate measurement mode (**Measure >> VSM Center Sample** in the MultiVu dropdown menu)

CAUTION!

Failure to perform frequent enough centering operations will lower the accuracy of measurements.

Automatic centering is a key quality-control mechanism in the software. When automatic centering has been disabled, the software cannot know how far the sample has drifted from the center of the pickup coils unless a touchdown is performed.

4.2.2 VSM Measurement Process: Synchronous Detection

To perform VSM measurements, the Model CM-A VSM motor module is programmed to oscillate the sample at the center of the coilset at a predetermined frequency and amplitude. The voltage induced in the coilset is then amplified by the preamp and detected by the Model CM-B

VSM detection module. The VSM detection module also reads the real-time sample position from the motor module.

The VSM detection module uses both the position and voltage signals to generate a complete and independent magnetization measurement every cycle of oscillation. In principle, a 40-Hz oscillation frequency can produce a 40-Hz data rate from the module. These data are averaged over an amount of time that you specify. For example, if you specify an averaging time of 1 second and the oscillation frequency is 40 Hz, the detection module would average the 40 readings per second into one reading per second.

To obtain an accurate measurement of the magnetic moment, the VSM software application uses calibration data from calibration files on the PC and from ROMs in the modules. The calibration data includes corrections for preamplifier gain errors, phase-shift errors, and the geometry of the coilset puck.

4.3 Installing a Sample

4.3.1 Attach a Sample and Measure the Sample Offset

1. Attach the sample to the sample holder using the techniques discussed in Chapter 3.
2. Use the sample-mounting station to measure the distance from the center of the sample to the bottom of the sample holder, reading the position from the scale as demonstrated in Figure 4-2. This distance is called the sample offset. Measure the sample position to an accuracy of 0.5 mm. The VSM hardware can safely accommodate a sample offset between zero (i.e., sample is at the bottom end of the sample holder) and 35mm. However, a sample offset less than ~30mm can result in a significant signal from the magnetic “end effect” of the sample holder. A sample offset larger than 35mm presents the risk of the sample holder rattling against the puck surface when measuring (see Sec. 4.2.1.1).

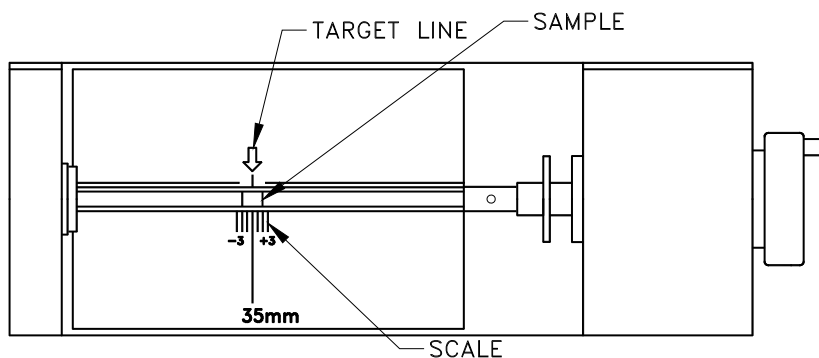


Figure 4-2. Reading the position of the sample from the sample-mounting station

3. Remove the sample holder (with the mounted sample) from the mounting station and screw it firmly onto the end of the sample rod.
4. Inspect the sample rod and sample holder to ensure they are straight. Deviations can result in rubbing of the sample or sample holder in the coil set which causes heating at low temperatures and noise in the measured moment when a magnetic field is applied.

4.3.2 Activate the VSM Option and Control Center

1. To start the VSM application and open the **VSM Control Center**, select **Utilities >> Activate Option** in the MultiVu window.
2. At this time, the system also will perform a **Home** operation to determine the full range of travel for the sample transport by touching down and then going to the load (top) position.

4.3.3 Install the Sample

1. In the **Install** tab of the **VSM Control Center**, click on the **Install/Remove Sample** button.
2. Click on the **Open Chamber** button (below the **Instructions** area). The wizard will bring the sample chamber to room temperature, vent the chamber, and move the transport to the load position, and the **Instructions** area will show the status of these processes.
3. When the **Instructions** area indicates that the chamber has been flooded and the transport is in the load position use the sequence outlined below to install the sample rod and sample.
 - If you have not already measured your sample offset position, use the sample-mounting station to obtain the sample offset from the end of the sample holder (see Section 4.3.1).
 - Attach the sample holder to the sample rod.
 - Remove the cap from the top of the VSM linear motor transport.
 - Insert the sample rod with the attached sample holder into the sample access port until the magnetic lock at the top of the sample rod engages the magnetic lock ring in the linear motor transport. Verify that the magnetic lock has engaged the magnetic lock ring.
Important: The sample will be subject to vertical magnetic fields of up to approximately 200 gauss when it passes through the head. If this is unacceptable for your samples, please contact your local Quantum Design service representative.
4. Click on the **Next >>** button at the bottom of the **VSM Install** wizard. If you have the VSM oven option then you will select Standard or Oven operating mode here,
5. Open a new or existing output data file. Note that all parameters entered here are for informational purposes only and are not used in calculating the reported sample moment.
6. Click on the **Next >>** button at the bottom of the dialog. Here you can **Scan For Sample Offset** or **Enter Offset Manually** if you used the sample mounting station to visually locate the sample.
 - A sample signal larger than $\sim 10^{-5}$ emu is required in order to be detected in the centering scan. This may require the application of magnetic field in order to magnetize your sample.
 - Magnetic contamination and sample holder end effects can obscure the sample signal, so it is recommended to verify the scan results with the known approximate sample offset of the sample.
 - The **Advanced Centering** mode will locate the motor at a fixed height and will not permit touchdown centering. Thus, it is not generally recommended.

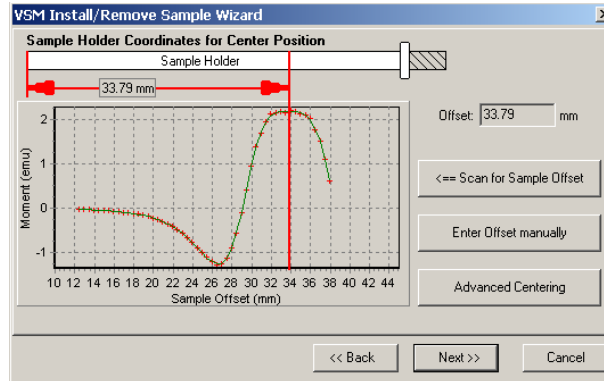


Figure 4-3. Example of a centering scan for a sample with a large positive magnetic moment of ~2 emu.

7. Click on the **Next >>** button at the bottom of the dialog (Figure 4-3). This button opens the last page of the **VSM Install** wizard (Figure 4-4), which reports the sample-offset position and related instructions.

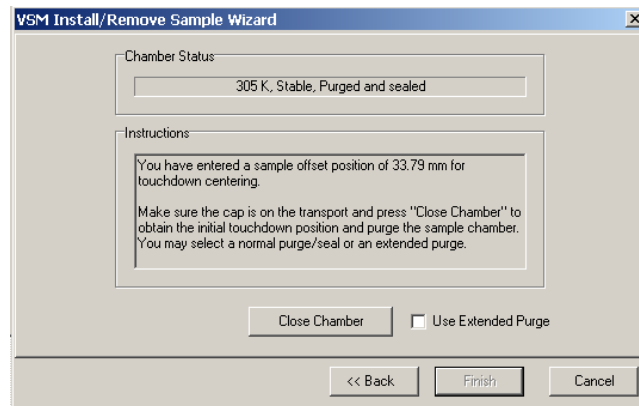


Figure 4-4. The VSM Install wizard, final page

Select **Use Extended Purge** any time you plan to use low temperatures ($T < 270$ K). Note that this ~10 minute extended purge must complete before measurements of temperature variations are started.

8. Place the cap on the linear motor transport and click on the **Close Chamber** button. **IMPORTANT:** do not perform any CPU-intensive tasks on the computer while the touchdown operation completes.
9. You can now set up the system to perform sample measurements.

4.4 Taking Immediate-Mode VSM Measurements

After the sample has been installed and the position of the sample has been specified, you can perform VSM measurements by using immediate-mode commands (e.g., from command buttons or dropdown menus) or by constructing sequence files. This provides a quick start guide for immediate-mode measurements. Chapter 6 describes sequence measurements and explains the parameters in the dialogs.

4.4.1 Measure Command and VSM Measurement Dialog

To specify parameters for VSM measurements, click on the **Measure** button, in the **VSM Control Center**. For most situations, the default settings for VSM measurement should be kept:

- **Continuous Measuring** will make best use of the fast lock-in based VSM measurement by streaming data continually.
- **Averaging Time** = 1 second will provide 10^{-6} emu noise levels and the speed will allow rapid field sweeping during measurements.
- **Logging Interval** = 0 will write all the measurements to the output data file instead of reducing the file size by only recording data at the stated interval.
- (In **Centering** tab) **Do Touchdown Centering at Intervals** is recommended in order to keep the sample close the center of the coil set.
- (In **Advanced** tab) Use **Peak Amplitude** = 2mm, default **Frequency** (usually 40 Hz), and **Sticky Autorange**

"Last Measurement" Area

The right side of each panel consists of the **Last Measurement** area, which displays the most recent measurement results. The displayed data are also written to the open data file along with the data items that are defined in the table in Chapter 6.

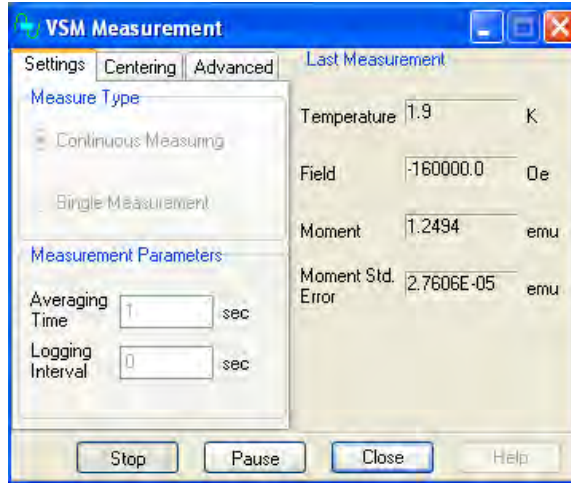


Figure 4-5. VSM Measurement dialog

VSM Hardware

5.1 Introduction

This chapter contains the following information:

- Section 5.2 describes each of the basic hardware components that make up the VSM option.
- Section 5.3 describes the contents of the VSM User's Kit.
- Section 5.4 describes the sample-mounting station and its use.
- Section 5.5 describes the electrical components of the VSM option.

5.2 VSM Hardware Components

This section describes each of the basic hardware components that make up the VSM system. For instructions about installing the various components, please refer to Chapter 2.

5.2.1 Linear Transport

The VSM linear transport (4096-400) is the motor that moves the sample. The linear motor transport and many of its component parts are illustrated in Figure 5-1. You will mount the linear motor transport directly on top of the PPMS sample-chamber opening after you have inserted the sample tube into the sample chamber.

The sample rod is inserted into the sample chamber through an access port on top of the VSM linear motor transport. The sample chamber is sealed (for storage and during operations) by a cap and an O-ring seal. An electrical connector at the rear of the linear motor transport provides the Model CM-A with electrical access to the drive coil, position encoder, and linear-motor-transport calibration ROM via the motor drive cable (3096-200 or 3096-201).

The sample rod is held in place in the VSM linear motor transport by a magnetic-locking mechanism consisting of small magnets in the top of the rod; the magnets stick to a thin steel ring at the top of the armature.

A spring-suspension mechanism inside the linear motor transport isolates the vertical motion of the motor from the housing during vibration. The resonant frequency of the spring-suspension mechanism is about 5 Hz. You can verify whether the suspension mechanism is functioning

correctly by gently tapping the top of the sample rod at the sample access port during sample loading. A mechanism that is working correctly will oscillate for at least 5 seconds.

The transport is shipped from the factory with the shipping plug installed to prevent oscillation of the motor suspension mechanism. Before using the equipment, you must remove the shipping plug and install the extender tube flange.

A window in the front side of the linear motor transport (opposite the electrical connector) displays the location of the armature (the moving section of the motor transport). When you are installing or removing samples, you will be able to see an indicator pin at the top of the window: The indicator pin will be at the *top* of the window when the system is in the “load” position. The indicator pin will be at the *bottom* of the window, which is the “shutdown” position, *only* when the sample rod has been removed and the linear motor transport has been shut down. During VSM operations, the indicator pin will vibrate rapidly (1–4 mm peak-to-peak amplitude at 40 Hz) between the load and shutdown positions.

Important: The sample will be subject to vertical magnetic fields of up to approximately 200 gauss when it passes through the head. If this is unacceptable for your samples, please contact Quantum Design.

CAUTION!

- ◆ The indicator pin should be at the bottom of the window when the linear motor transport has been shut down. If it is NOT, it indicates that the sample rod has been inadvertently left in the sample chamber.
- ◆ Never attempt to remove the linear motor transport from the system while the sample rod is installed.

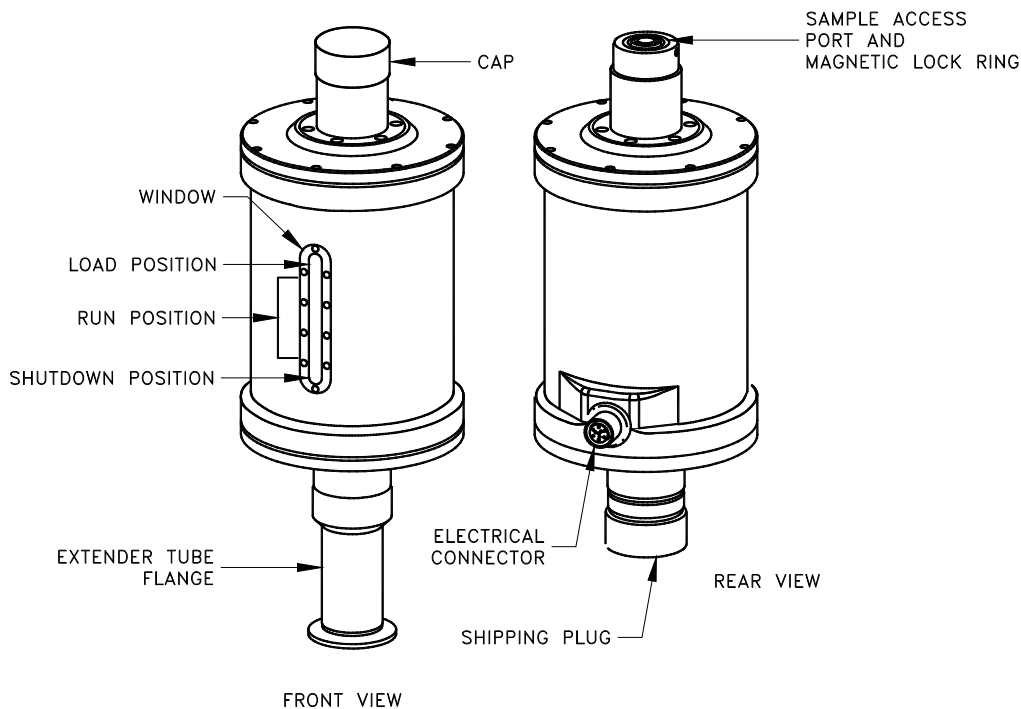


Figure 5-1. VSM linear motor transport (4096-400)

5.2.2 Storage Case for Linear Motor Transport

Because the linear motor transport is both heavy and delicate, Quantum Design has furnished you with a specially designed storage case to protect it during transport and storage. When the linear motor transport is not being used, it should be stored in this case. You can keep the case on a shelf or in a cabinet, so long as it is on a stable base and is stored upright as shown in Figure 5-2.

CAUTION!

Because the storage case has a high center of gravity, it is critical that you place it on a very stable base such as the floor.

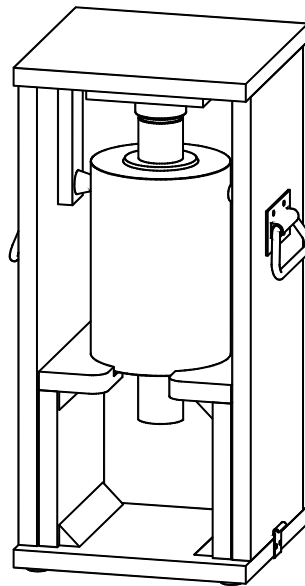


Figure 5-2. Storage case (4096-150) for the linear motor transport.

5.2.3 Coilset Puck

The coilset puck contains the VSM detection coils and a thermometer for monitoring the sample temperature. You will insert the coilset puck into the sample chamber by using the PPMS sample insertion tool (also called the puck insertion/extraction tool) and the same procedures used to insert other types of pucks (see the *Physical Property Measurement System: Hardware Manual* for information on puck insertion and extraction). You must install the puck **before** you insert the sample tube.

The electrical connector at the bottom of the coilset puck has a serial number (see Figure 5-3). As explained in Sections 2.2.3, you will use this serial number to verify the numbers contained in the application software. The system uses the serial number to identify the puck calibration information.

The VSM option is flexible enough to accommodate different detection-coil configurations—all you will need to do is change the coilset puck. However, for most uses, the standard coilset puck provides the best trade-off between sensitivity and accuracy.

The dimensions of the standard coilset puck are shown schematically in Figure 5-4. The center of the gradiometer pick-up coils is located 40.1 mm (1.58 in) above the location corresponding to the puck surface. This position corresponds to the center position of PPMS high-field systems (14-T or greater) and the high homogeneity region of lower field magnets (e.g., 7-T, 9-T).

After repeated insertions of the VSM puck, the contact fingers at the base of the coilset (above the serial number in Figure 5-3) might bend inwards, which would loosen the fit of the coilset in the bottom of the sample chamber. When this happens, you should make the coilset fit snugly again by using the puck adjustment tool, as described in the *Physical Property Measurement System: Hardware Manual*.

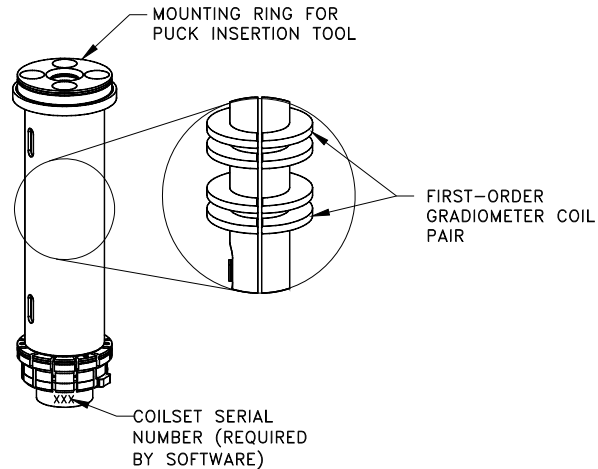


Figure 5-3. Coilset puck (4096-204)

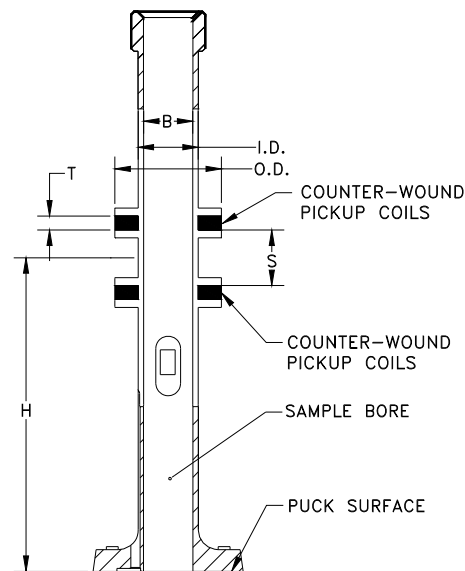
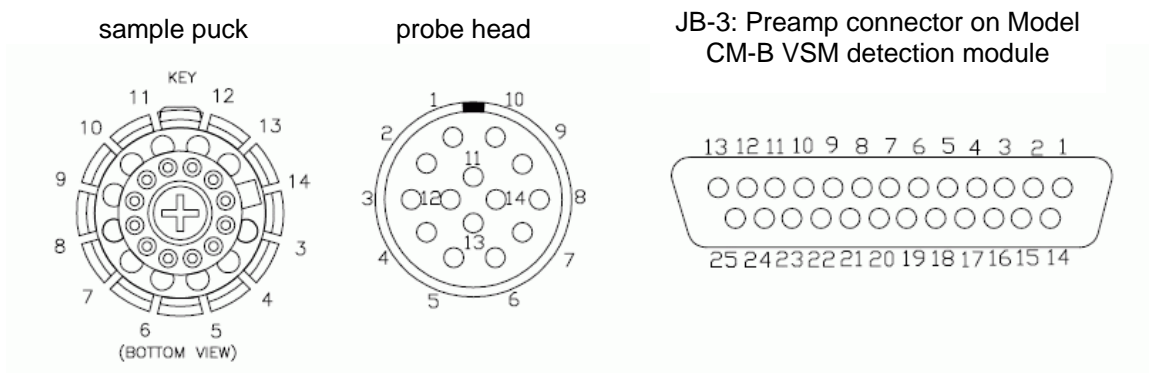


Figure 5-4. The internal dimensions of a standard coilset puck are as follows: Bore diameter (B) = 6.33 mm; coil thickness (T) = 1.78 mm; coil spacing (S) = 7.11 mm; coil inner diameter (ID) = 7.73 mm; coil outer diameter (OD) = 13.7 mm; height above puck surface (H) = 40.1 mm.

Table 5-1. Sample Connection with VSM Detection Cable Connected. Note that pins 7-10 on the sample puck are unused. Also, the detection coilset is connected to Channel 1 (puck pins 11 and 12).



SAMPLE PUCK	GRAY LEMO CONNECTOR AT PROBE HEAD	JB-3 PREAMP ON CM-B VSM MODULE	FUNCTION
3	3	5	Thermometer Current +
4	4	18	Thermometer Current -
5	5	6	Thermometer Voltage +
6	6	19	Thermometer Voltage -
11	11	10	Channel 1 Input +
12	12	23	Channel 1 Input -
13	13	12	Channel 2 Input +
14	14	25	Channel 2 Input -

5.2.4 Sample Tube

You will insert the sample tube (Figure 5-5) into the sample chamber after you have installed the coilset puck. The sample tube provides low-friction guide sleeves for the sample rod. The top of the sample tube consists of an integrated O-ring attached to a stabilizer post. The stabilizer post provides a rigid, bayonet-style mount that prevents the linear motor transport from tipping over. Part of the post extends into the sample chamber and part extends into the extender tube flange on the bottom of the linear motor transport, as shown in Figure 2-7.

CAUTION!

You should always use the flange clamp to hold the linear motor transport onto the post, even though the stabilizer post helps prevent the linear motor transport from tipping over.

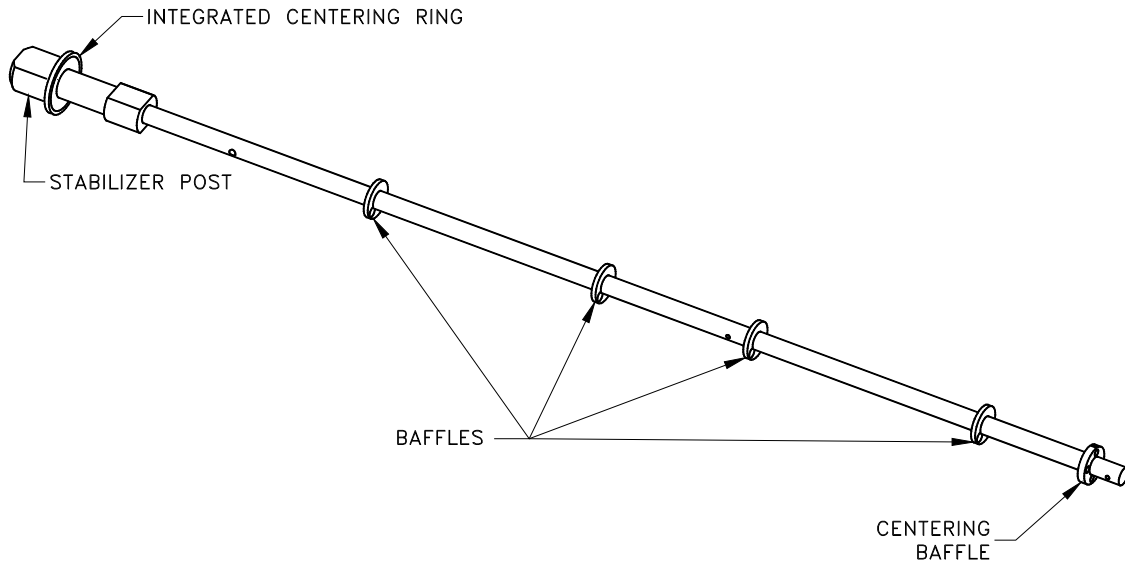


Figure 5-5. VSM sample tube (4096-301 shown for example)

5.2.5 Sample Rod

Many of the design features of the sample rod were introduced to reduce the background signal from mechanical vibrations at the VSM oscillation frequency. Such features control the rattling or friction between the rod and the system and they eliminate resonant flexure of the shaft.

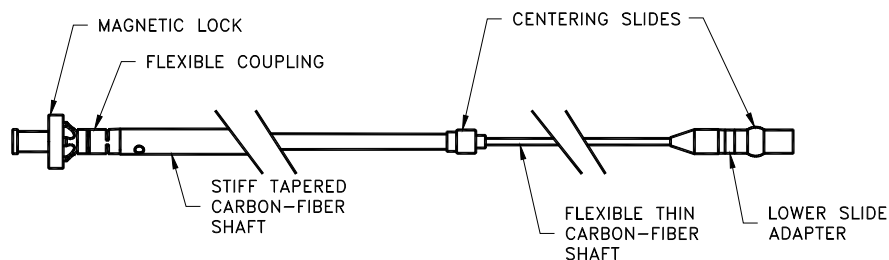


Figure 5-6. VSM sample rod (4096-352 shown for example)

Figure 5-6 shows the sample rod in a horizontal position (during use, the magnetic lock would be at the top of the rod).

Important: Keep the magnet surfaces clean, as the strength of the lock depends on the magnets being flush in contact with the mating part in the linear motor transport. Also, avoid bringing the magnets into contact with magnetic objects. Although small, the magnets are extremely strong.

5.3 VSM Option User's Kit

The VSM Option User's Kit contains miscellaneous hardware and supplies that you will use to mount samples and to verify the operation of the VSM. The convenient portable toolbox (see Figure 5-7) helps organize the items.

The kit includes the following contents:

- **Sample-Mounting Station**

The sample-mounting station is used to mount samples at the correct location in the sample holder. For more information about mounting samples, see Figure 5-8 and Section 3.3.

- **Sample Holders**

Sample holders are used to hold samples (see Figure 5-8); the holders screw onto the bottom of the VSM sample rod. Quantum Design has provided five paddle-shaped sample holders and five trough-shaped sample holders.

- **Calibration Sample**

The palladium calibration sample is used to calibrate the VSM option and to verify its accuracy.

- **Coilset Assembly**

The VSM coilset assembly is shipped as part of the VSM Option User's Kit.

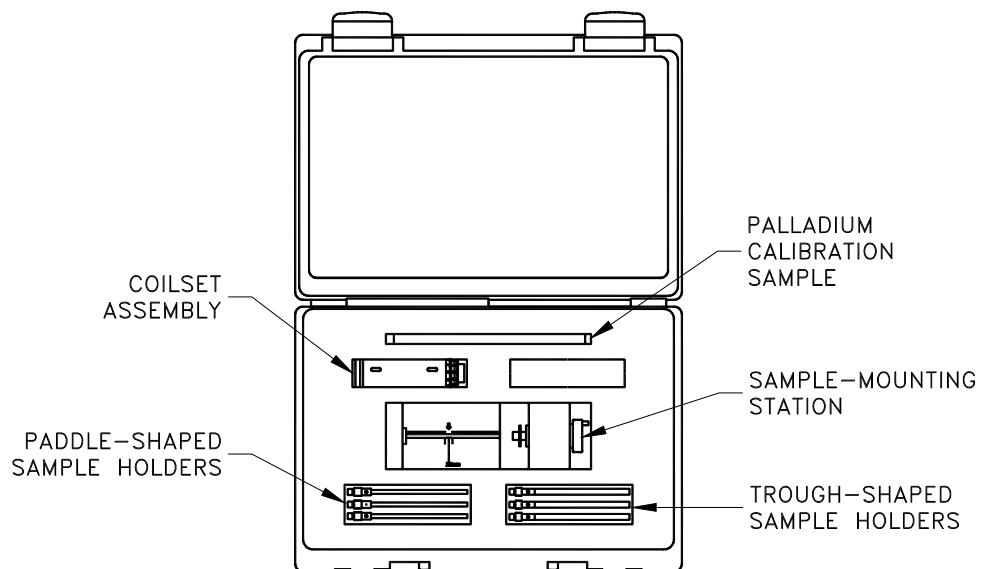


Figure 5-7. VSM Option User's Kit (4096-100)

5.4 Sample-Mounting Station

The sample-mounting station (4096-110) is used to precisely locate and measure (within 0.5 mm) the sample offset, which is the distance between the sample and the bottom of the sample holder (see Figure 5-8).

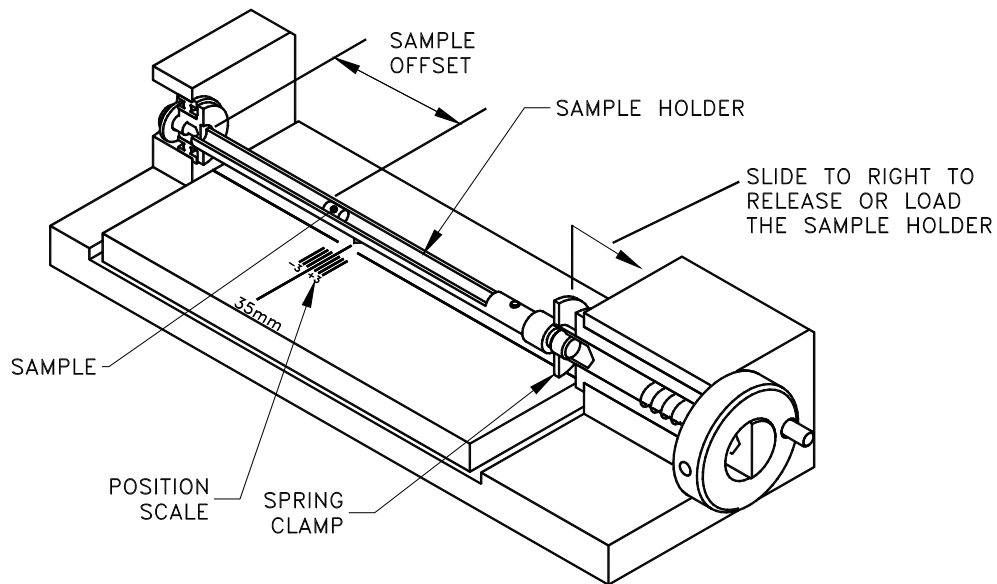


Figure 5-8. Sample-mounting station (4096-110)

The VSM measurement algorithm requires an accurate measurement of this distance so that it can perform touchdown operations (See Chapters 1 and 4 for more details about touchdown operations).

5.5 VSM Electronics

5.5.1 Preamp Cable Assembly

Figure 5-9 shows the preamp cable assembly (3096-300), which is the electrical connection between the coilset puck and the “PREAMP” port (JB-3) on the Model CM-B detection module. Integrated into the cable are dual 40-gain preamplifiers for up to two independent detection coils. The preamp end of the cable is plugged into the gray connector at the PPMS probe head.

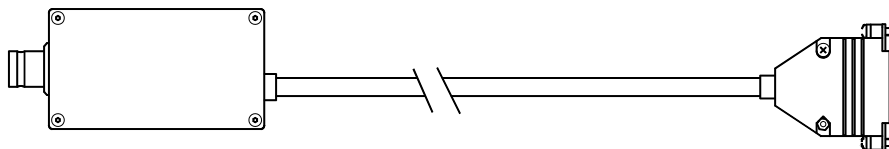


Figure 5-9. Preamp cable assembly (3096-300)

5.5.2 Motor Drive Cable

Figure 5-10 shows the VSM motor drive cable (3096-200), which connects the back of the linear motor transport to the “SERVO” port (JA-1) on the Model CM-A (motor module). The drive cable provides drive-coil power to the linear motor transport, position-encoder read-back to the module, and serial communication between the linear motor transport and the module for diagnostic and configuration purposes.

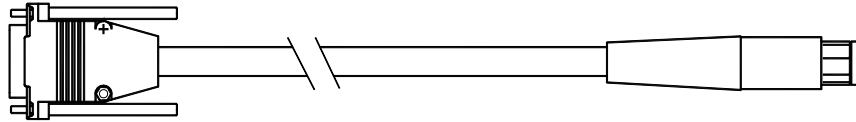


Figure 5-10. Motor drive cable (3096-200)

5.5.3 Model CM-A (VSM Motor Module)

Figure 5-11 shows the Model CM-A VSM motor module (4101-100), which provides all the power and logic that are necessary to drive the VSM linear motor transport. All configuration and control of this module is through the VSM application software on the computer (PC) via the CAN-bus connector on the back panel of the module.

See Appendix A for a detailed description of the functions of this module.

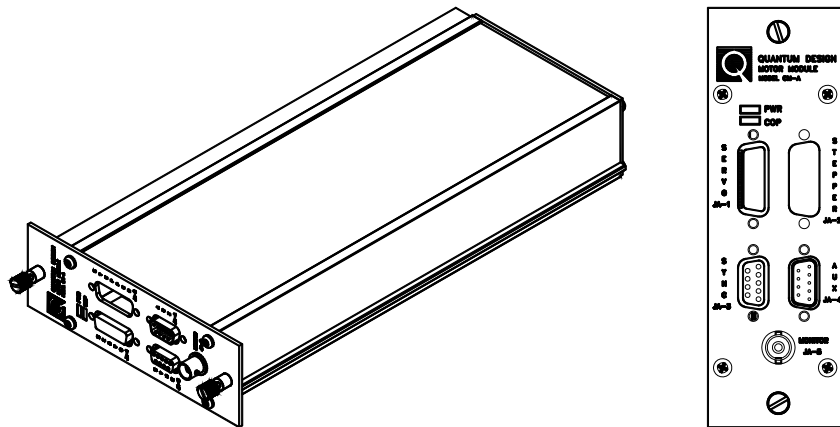


Figure 5-11. Model CM-A VSM motor module (4101-100)

5.5.4 Model CM-B (VSM Detection Module)

Figure 5-12 shows the Model CM-B (4101-150), which performs the synchronous detection (or “lock-in”) operation for the VSM option. The reference position signal comes from the Model CM-A VSM motor module via the VSM–motor sync cable; the induced voltage signal from the coilset puck comes via the preamp cable assembly. All configuration and control of this module is handled through the VSM application software on the computer (PC) via the CAN-bus connector on the back panel of the module.

See Appendix B for a detailed description of the functions of this module.

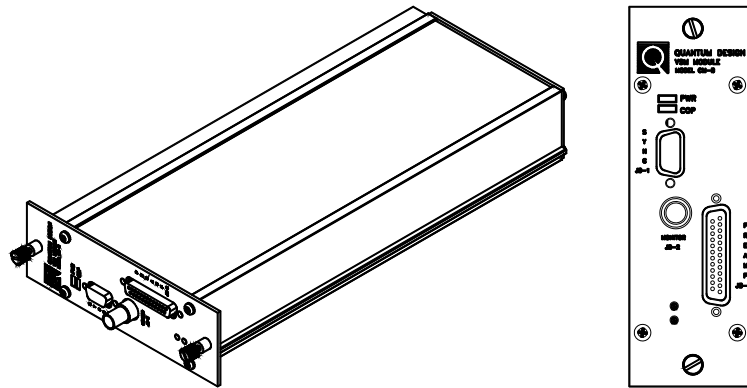


Figure 5-12. Model CM-B VSM detection module (4101-150)

5.5.5 Model 1000 (Modular Control System) (PPMS ONLY)

The Model 1000 modular control system (4100-001) is a general-purpose chassis (Figure 5-13) that houses, cools, and provides power to both the Model CM-A VSM motor module and the Model CM-B VSM detection module. The Model 1000 can accommodate up to four additional modules for other PPMS options. The backplane provides connections for power as well as a CAN-based network connection to the computer (PC).

See the *Model 1000 Modular Control System User's Manual* for more information about the Model 1000 modular control system.

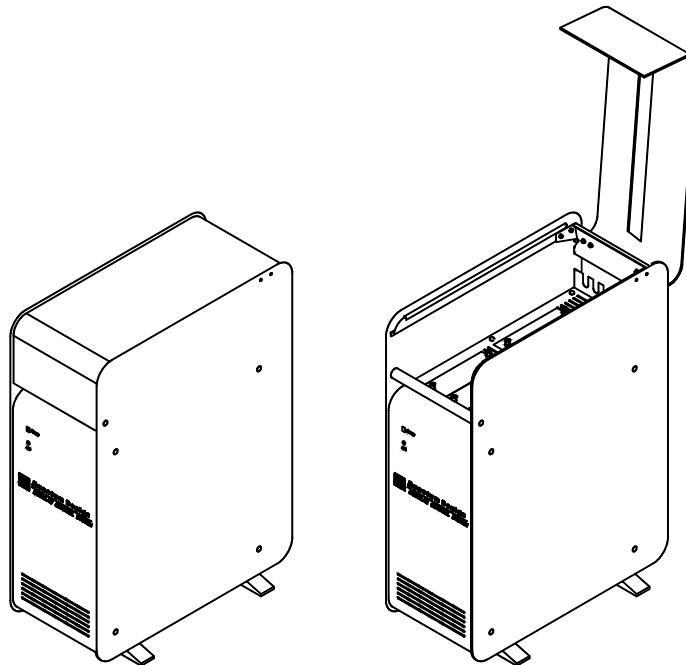


Figure 5-13. Model 1000 modular control system (4100-001)

5.5.6 CAN Computer Interface Kit

The CAN computer interface (or network adapter) kit contains the CAN-based network adapter, cable (3100-024), and software that are needed to connect the Model 1000 to the computer (PC).

For measurement platforms such as DynaCool and VersaLab, the CAN Computer Interface is built into the instrument.

VSM Software

6.1 Introduction

This chapter contains the following information:

- Section 6.2 describes option activation and deactivation.
- Section 6.3 describes the VSM-specific dialogs and menu items in the MultiVu dropdown menus.
- Section 6.4 describes the **VSM Control Center** and its components.
- Section 6.5 briefly describes sequence mode commands.
- Section 6.6 explains use of the VSM sequence-mode **Adv. Measure** command.
- Section 6.7 explains use of the VSM sequence-mode **Center Sample** command.
- Section 6.8 explains use of the VSM sequence-mode **Moment vs. Field** command.
- Section 6.9 explains use of the VSM sequence-mode **Moment vs. Temp** command.
- Section 6.10 describes the format of VSM data files.

6.2 Activating and Deactivating the VSM Option

1. To activate the VSM application and open the **VSM Control Center**, select **Utilities >> Activate Option** from the dropdown menu of the MultiVu window (Figure 6-1).

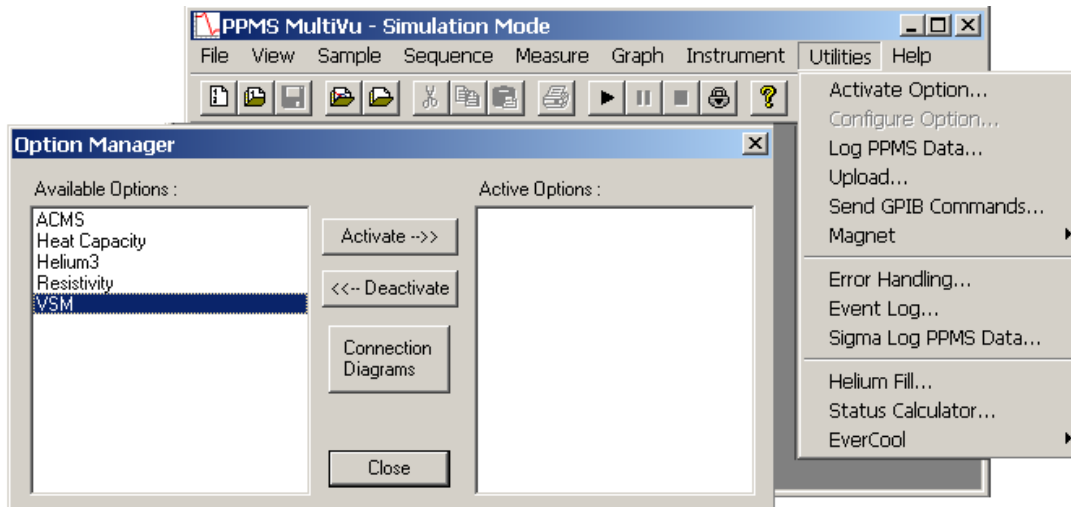


Figure 6-1. Using the MultiVu dropdown Utilities menu and Option Manager to activate the VSM Option

2. When the **Option Manager** dialog opens, select **VSM** and click on the **Activate** button. This will move the VSM option from the **Available Options** section of the dialog to the **Active Options** section. The **VSM Control Center** and the **VSM Log** window will open concurrently. When running in simulation mode, the **VSM Control Center** is titled **VSM SIM** (see Figure 6-2). To deactivate the VSM option, select it on the right side and click **Deactivate** in this dialog.

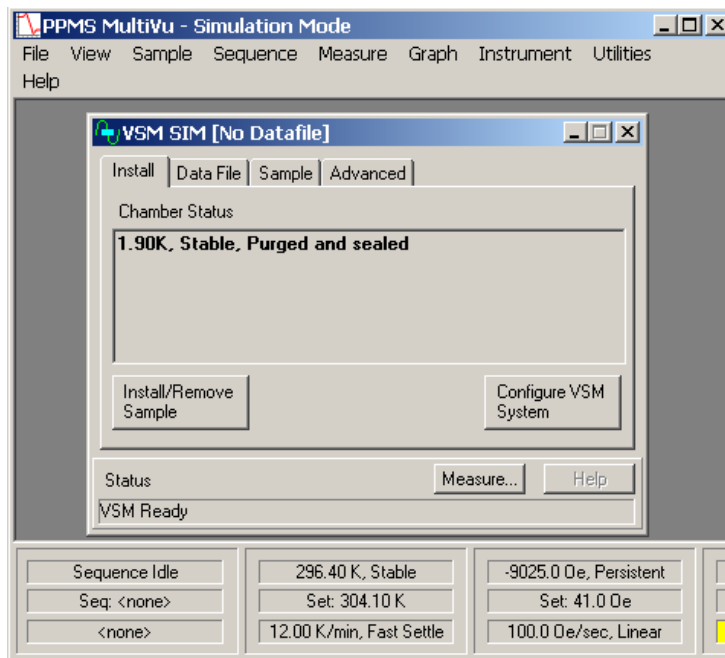


Figure 6-2. The MultiVu window and the VSM Control Center (VSM SIM), including the Install tab, the VSM Status area, and the MultiVu Status bar. Note that the VSM Log window has been minimized.

6.3 VSM Dropdown Menus

VSM-specific actions, dialogs, and commands are incorporated into the **View**, **Sample**, and **Measure** dropdown menus in the MultiVu window after you have activated the option. The other dropdown menus in the MultiVu window contain menu items that are common to all Quantum Design systems.

6.3.1 View

You can use the **View** dropdown menu at the top of the MultiVu window (Figure 6-3) to open VSM-specific dialogs as well as to perform general actions, such as opening and closing the MultiVu **Status Bar**, the **Sequence Control** bar at the side of the window, or the tool bar.

- Select **VSM Status Log** to view the events in the VSM log since the option was activated. The file located in \VSM\Logfiles\VSMlog.txt contains the entire history of this log and is a useful resource for troubleshooting.
- Select **VSM Error Count** to open a dialog showing only errors encountered as well as the total count.

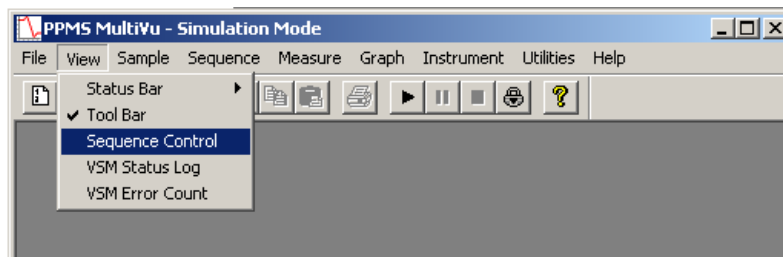


Figure 6-3. The MultiVu window and the View dropdown menu showing VSM immediate-mode commands

6.3.2 Sample

You can use the **Sample** dropdown menu at the top of the MultiVu window (Figure 6-4) to install and remove samples and to center the sample manually.

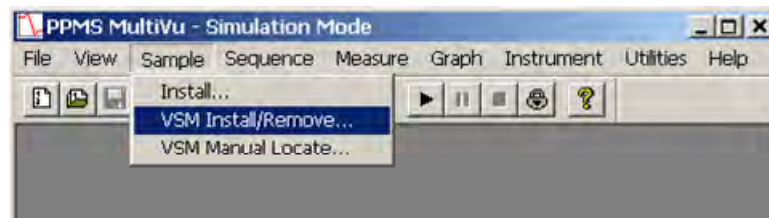


Figure 6-4. The MultiVu window and the Sample dropdown menu showing VSM immediate-mode commands

Important: The **Sample** dropdown menu contains two "install" selections: **Install...** and **VSM Install/Remove ...** You should always select **VSM Install/Remove...** when using the VSM. The **Install...** selection applies to the base system and is not appropriate for the VSM option.

- To install a sample, designate an output data file, and center the sample, select **Sample >> VSM Install/Remove**. This selection opens the **VSM Install/Remove Sample Wizard** (Figure 6-5).
- To provide the software with the location of the sample, select **Sample >> VSM Manual Locate**. The **Specify Sample Location** dialog will open so that you can enter the sample offset or perform an automatic scan. This dialog also appears when you use the **VSM Install/Remove Sample Wizard** to install a sample (Section 4.3.3).

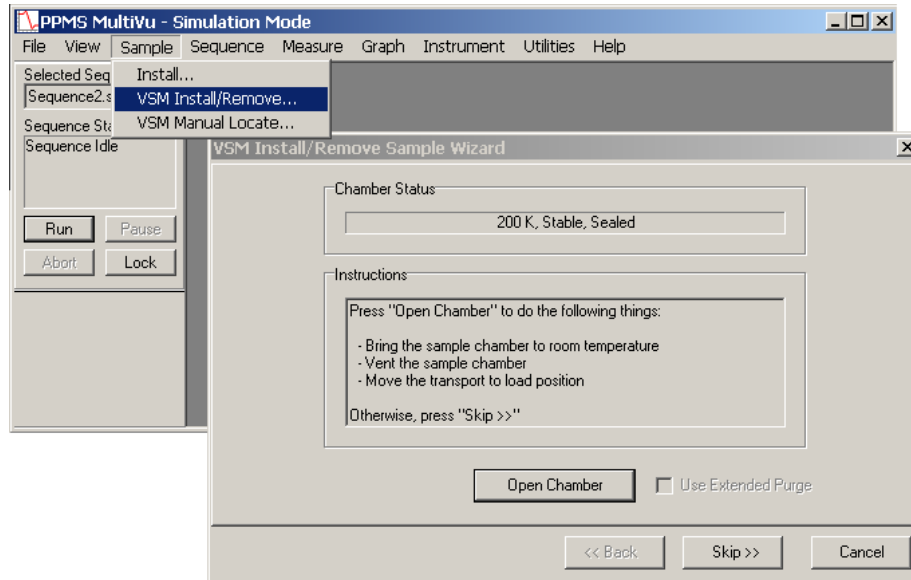


Figure 6-5. The Specify Sample Location dialog that opens from the dropdown menu when you select Sample >> VSM Manual Locate

6.3.3 Measure

If a sample has been installed in the VSM, you can use the **Measure** dropdown menu at the top of the MultiVu window (Figure 6-6) to open immediate-mode VSM measurement dialogs (e.g., for setting up and taking measurements, for centering the sample, and for adding a comment to an output data file). In the event you attempt to use commands in the **Measure** dropdown menu but you have not installed a sample, a popup message will open, directing you to first install a sample.

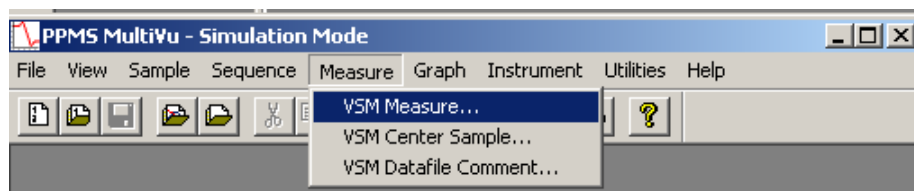


Figure 6-6. VSM immediate-mode commands in the context of the MultiVu window and the Measure dropdown menu

- To set up and take measurements, select **Measure >> VSM Measure**. The **VSM Measurement** dialog will open (Figure 6-14), with three tabs (or pages) of settings you can use to delineate your measurements. The same dialog appears when you click on the **Measure** button in the **VSM Control Center**. For a complete explanation of the **VSM Measurement** dialog, see Section 6.4.2.

- To center a sample, select **Measure >> VSM Center Sample**. This selection sequence opens the **VSM Center Sample** popup (Figure 6-7), which allows you to initiate touchdowns at will. As explained in Chapter 4, centering operations help ensure the accuracy of measurements.
- To add a comment to a VSM data file, select **Measure >> VSM Datafile Comment**. When the **VSM Datafile Comment** popup appears (Figure 6-8), enter your comment in the text box. The text will be appended to the currently open VSM data file in the Comment column (first column).

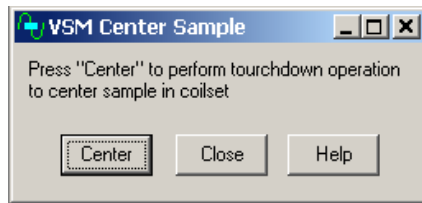


Figure 6-7. The immediate-mode VSM Center Sample popup

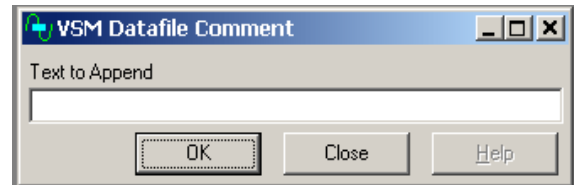


Figure 6-8. The immediate-mode VSM Datafile Comment popup

6.4 VSM Control Center

The **VSM Control Center** opens within the MultiVu window as soon as you activate the VSM option and application software. Figure 6-9 shows the **VSM Control Center** and the commands, dialogs, tabs, buttons, and software prompts that organize measurement-related activities, making it easy for you to perform basic operations (e.g., create data files, install samples, and set up and initiate immediate-mode measurements).

6.4.1 VSM Control Center: Components

6.4.1.1 INSTALL TAB

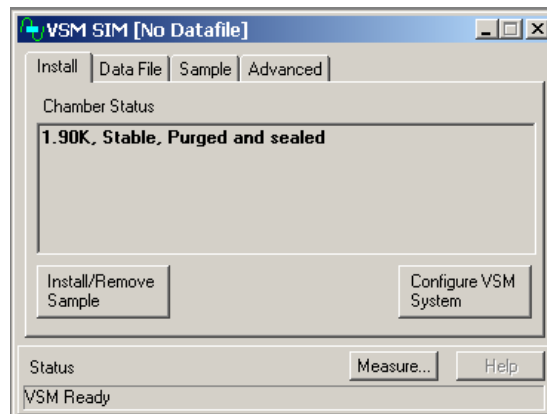


Figure 6-9. Install tab in the VSM Control Center

The **Install** tab provides access to two wizards.

- **Install/Remove Sample** guides the user through installation of a sample into the VSM measurement space and is outlined in Section 4.3.3.
- **Configure VSM System** will allow the user to select a different active VSM coilset (see Section 2.2.8). Coilsets are distinguished by serialization on the puck base (see Section 5.2.3) and there is a corresponding calibration file VSMxxx.CFG in the VSM/Calibration folder. One sign that an incorrect coilset is in use is that the temperature between coilset and base system differ by >1% or the VSM option declares that the option thermometer is not responding.

6.4.1.2 DATA FILE TAB

The Data File tab (Figure 6-10) identifies the output data file that will contain the measurement data. If you have not selected an output data file, the File Name and Title panels in the Data File tab will be blank.

View Button

The **View** button opens the graph view of the active output data file. To use the **View** button, you must have designated an output data file.

Browse... Button

The **Browse...** button initiates a series of dialogs that guide you through the process of designating (selecting or creating) a data file. The file-designation process and the dialogs are similar to the ones in the **Install/Remove Sample Wizard** (see Section 4.3.3).

Important: You must designate an output data file (by selecting an old file or by making a new one) if you want the measurement data saved. If no data file is designated, the measurement data will be written to the file `\Data\VSMdefault.DAT`.

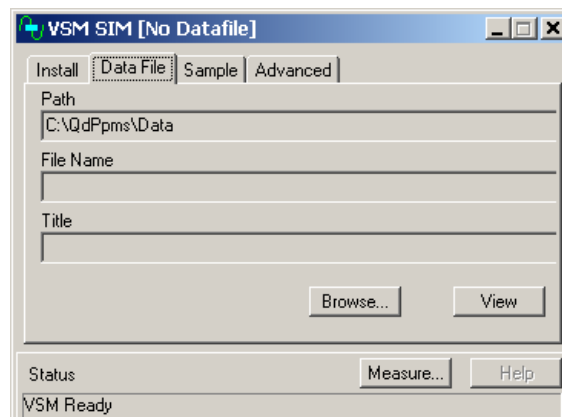


Figure 6-10. Data File tab in the VSM Control Center

Creating a new data file will prompt the user to enter sample properties as shown in Figure 6-11. This information, recorded in the header of the data file, is for information only and is not used in calculating the reported magnetic moment.

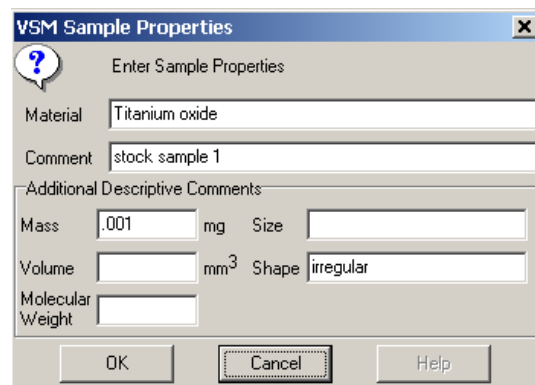


Figure 6-11. Recording sample properties

6.4.1.3 SAMPLE TAB

The **Sample** tab (Figure 6-12) is a read-only status display that shows the sample properties (e.g., material, comments, mass, and volume) for the active output data file. The information in the **Sample** tab originates from your entries in the **VSM Sample Properties** dialog, which are stored in the header of the output data file. If you select a pre-existing file to which you want data appended and bring the **Sample** tab forward, the information in the **Sample** dialog will reflect the entries in the header of the pre-existing file.

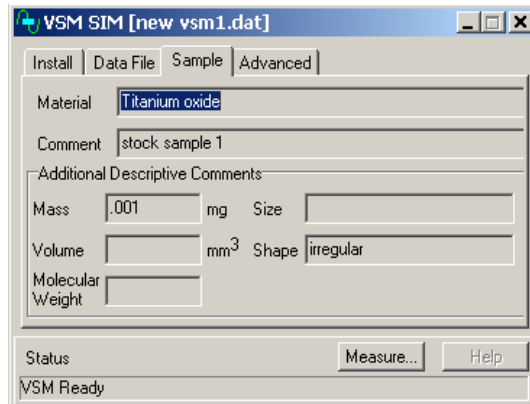


Figure 6-12. Sample tab in the VSM Control Center

6.4.1.4 ADVANCED TAB

The **Advanced** tab (Figure 6-13) organizes support for troubleshooting, for example, checking calibrations and troubleshooting system performance. Hence, you will use the options on the **Advanced** tab only if you are an experienced VSM operator.

- The **Units of Measure** options allow you to record in emu or in A-m² (ampere meters squared). By default, the measures are recorded as emu.
- The **Motor Friction Scan** button moves the VSM linear motor transport through the full range of motion. The system plots the motor force (as current) as a function of position for both lifting the sample and lowering the sample. A difference between these two curves indicates friction due to ice or other obstructions.

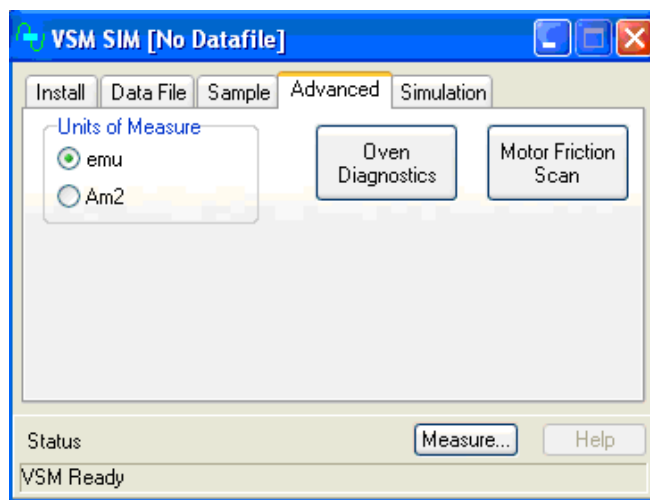


Figure 6-13. Advanced tab in the VSM Control Center

6.4.2 VSM Control Center: "Measure" Button

The **Measure** button in the **VSM Control Center** opens the **VSM Measurement** dialog box (Figure 6-14), which organizes immediate-mode measurement settings and initiates measurements. The dialog has a section showing the measurements, three tabs (**Settings**, **Centering**, and **Advanced**), and three buttons (**Start (Stop)**, **Pause (Resume)**, and **Close**).

6.4.2.1 "VSM MEASUREMENT" DIALOG: BUTTONS

- The **Start (Stop)** toggle button starts and stops the measurement process, but it does not close the dialog.
- The **Pause (Resume)** toggle button only stops the system from performing measurements, as the system continues to oscillate the sample and execute any scheduled automatic touchdown operations. To restart the data output after a **Pause**, click on the **Resume (Pause)** toggle button.
- The **Close** button closes the **VSM Measurement** dialog, but it does not stop the measurement.

6.4.2.2 "VSM MEASUREMENT" DIALOG: LAST MEASUREMENT

The **VSM Measurement** dialog is divided into two main areas: On the left side are the tabs with the measurement settings. On the right side is a **Last Measurement** area that displays the most recent **Temperature**, **Field**, **Moment**, and **Moment Std. Error**. **Error** data, which are written to the open data file.

- **Temperature** represents the average temperature during the measurement, in Kelvin.
- **Field** represents the average field, in oersted.
- **Moment** represents the average of the moment over the averaging time, in emu.
- **Moment Std. Error** represents the error on the mean, that is, the error bar on the reported moment.

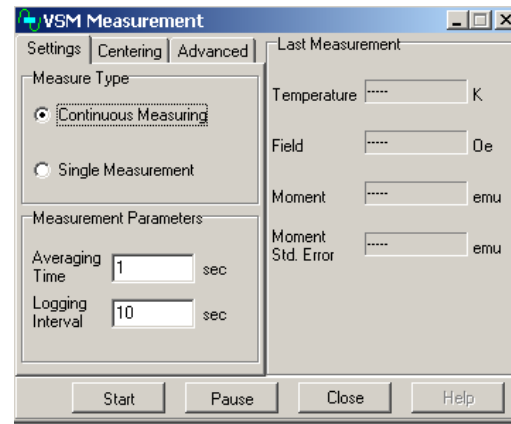


Figure 6-14. VSM Measurement dialog and the Settings tab

6.4.2.3 "VSM MEASUREMENT" DIALOG: SETTINGS TAB

The **Settings** tab (Figure 6-14) is divided into options for **Measure Type** and **Measurement Parameters**.

Measure Type

Measurements can be taken in **Continuous** mode in which new data is collected and written to the data file until the **Stop** button is pressed. The **Single Measurement** mode collects one data point upon pressing the **Start** button.

Measurement Parameters

- **Averaging Time** specifies the boxcar average duration for each measurement and will be an integer number of periods of sample oscillation. A practical minimum for this time interval is 0.5 seconds and a typical value is 1 second.
- **Logging Interval** is the amount of time that elapses between recorded measurements, in seconds. If you set **Logging Interval** to a value less than **Averaging Time**, then one data

6.4.2.4 "VSM MEASUREMENT" DIALOG: CENTERING TAB

The **Centering** tab (Figure 6-15) displays the automatic centering settings that are in effect. Normally, you will not need to adjust these settings.

It is recommended to use touchdown centering to ensure the sample remains centered in the coil set. See the discussion in Sec. 7.4 regarding artifacts that can arise when **No Automatic Centering** is selected.

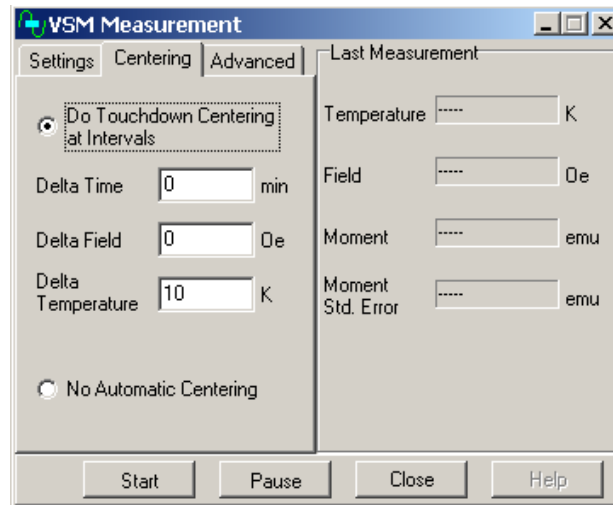


Figure 6-15. Centering tab in the VSM Measurement dialog

Touchdown operations will occur when any one of the monitors (time, field or temperature) has been exceeded. The default parameters of Delta Time = 10 minutes and Delta Temperature = 10 K will be sufficient for most cases. Selecting zero (0) for any of the monitors will turn it off. For instance, Delta Field = 0 should be used for all systems except the 16 tesla PPMS (it is provided because the stray field of the main magnet can slightly pull on the motor and change the operating height of the sample).

Touchdowns result in a approximately 20 second interruption in measurements. If this is unacceptable in your measurements then centering can be triggered explicitly by selecting **Measure >> VSM Center Sample** or using the **Center Sample** sequence command.

6.4.2.5 "VSM MEASUREMENT" DIALOG: ADVANCED TAB

The **Advanced** tab of the **VSM Measurement** dialog (Figure 6-16) contains settings for **Excitation Parameters**, **Ranging**, and **PPMS Data Logging**.

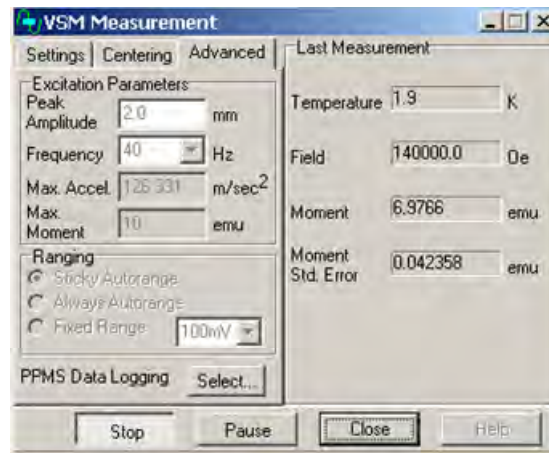


Figure 6-16. Advanced tab in the VSM Measurement dialog

Excitation Parameters

The excitation parameters are explained by the equation:

$$\text{Position}(\text{time}) = [\text{Peak Amplitude}] \times \sin(2\pi \times [\text{Frequency}] \times \text{time})$$

- The **Peak Amplitude** is typically set to 2 mm and can be varied from 0.1 to 5 mm with a recommended range of 0.5 to 4 mm. Low amplitudes allow for measuring a larger moment (see **Max Moment** value in this dialog) because the induced coil set signal is proportional to the amplitude. However, low amplitudes (<0.5mm) can result in an inaccurate reported moment. High amplitudes provide more coil set signal but also produce large accelerations (see **Max Accel.** value in this dialog) which can lead to higher noise in measurements.

Important: Quantum Design staff recommend that you limit the maximum **Peak Amplitude** to 4 mm, because the motor module could overheat at greater amplitudes. By limiting the maximum **Peak Amplitude** to 4 mm, you also ensure that the sample holder clears the puck surface. For example, taking into account that the detection coils are 40 mm above the puck surface and using a sample offset of 35 mm, the use of a **Peak Amplitude** of 5 mm or greater would cause the sample holder to touch the puck surface, which could dislodge the sample or the sample rod.

- The **Frequency** is typically 40 Hz; it specifies the frequency with which the VSM oscillates the sample. It can be moved to a different value if there is interference at 40 Hz. See PPMS Service Note 1096-304 at www.qdusa.com for information about changing the vibration frequency.
- **Max. Accel.** is computed from **Frequency** (40 Hz) and the **Peak Amplitude** entry; it represents the maximum acceleration the sample will experience during a measurement in units of meter per second squared.

Important: Do not proceed with a measurement if your sample cannot tolerate accelerations of this magnitude. To reduce the acceleration, reduce the amplitude.

- **Max. Moment** is computed from **Frequency** (40 Hz) and the **Peak Amplitude** entry; it represents the maximum sample moment that can be measured using these settings.

Important: Do not proceed with a measurement if the magnetic moment of your sample is larger than the calculated **Max. Moment**, as the system will not be able to complete the measurement. Larger moments can be measured by using relatively small values for amplitude.

Ranging

The **Ranging** setting refers to the way the system chooses the gain of the amplifiers in the VSM module during a measurement; the optimal setting is typically **Sticky Autorange**. The preamplifiers in the VSM module can change the gain ranges by factors of 10, depending on the size of the signal that is induced in the pickup coils. In the rare case when you need more control than is offered by **Sticky Autorange**, you can change the **Ranging** setting.

- **Sticky Autorange** (recommended): The system automatically increases the gain by a factor of 10 if the current peak signal drops below 2% of the current range. The system automatically decreases the gain by a factor of 10 if the current peak signal exceeds 50% of the current range.
- **Always Autorange**: The system automatically increases the gain by a factor of 10 if the current peak signal drops below 9% of the current range. The system automatically decreases the gain by a factor of 10 if the current peak signal exceeds 100%.

In some cases, the "**Always Autorange**" setting might improve the signal-to-noise ratio. However, this setting could also lead to an increase in the number of range changes and a corresponding drop in data throughput.

- **Fixed Range**: The system always uses the specified gain range. This can be useful when measuring samples which change signal rapidly (e.g., ferromagnets with a very sharp hysteresis loop) and range-changing is not desired.

Data Logging

In the **Data Logging** area of the **VSM Control Center Advanced** tab, the **Select ...** button opens the dialog (Figure 6-17), which lists additional system data items that you can choose to add to the current data file. These items are in addition to the VSM data items that are typically included, which are summarized in Table 6-1.

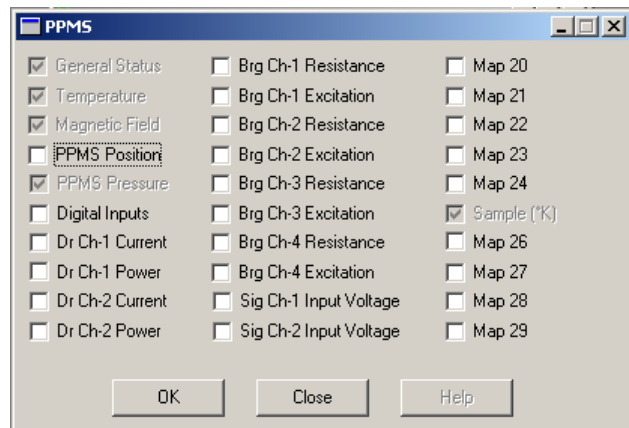


Figure 6-17. Data-logging dialog for selecting additional types of data to be collected

6.5 Overview of Sequence-Mode VSM Commands

VSM sequence-mode commands are, essentially, encapsulated versions of VSM immediate-mode measurement commands. **VSM Adv. Measure**, **Center Sample**, **Moment vs. Field**, and **Moment vs. Temp.** sequence commands can be combined with non-VSM sequence commands and looping constructs. The VSM sequence-mode commands have interactive dialogs that help you specify your measurements, and these dialogs are similar to the ones used to set up the immediate-mode measurements.

6.6 Sequence-Mode VSM "Adv. Measure" Command

When you select and click on the sequence-mode **Adv. Measure** (advanced measure) command, the sequence-mode **VSM Measurement** dialog opens so that you can specify the measurement parameters. These dialogs mirror those of the immediate mode measurements described above so they will not be repeated here.

6.7 Sequence-Mode VSM "Center Sample" Command

If you choose not to use automatic centering, you will need to perform centering operations by using the dropdown menu or by inserting centering operations into a sequence file. As explained in Chapter 4, centering operations help ensure the accuracy of measurements, keeping the centering position stable to within about 0.1 mm at the center of the pickup coils by informing the system about sample position shifts with respect to the coilset.

To insert a centering operation into a sequence file, click on the **Center Sample** sequence command in the **Sequence Commands** bar. This opens the **VSM Center Sample Sequence** dialog shown in Figure 6-18.

When the sequence file is run and the program encounters the **Center Sample** sequence command, it temporarily halts the current **VSM Adv. Measure** command. During the pause, the program performs a centering operation and adjusts the center of oscillation for the measurement. Then it resumes the measure command, continuing from the point where it stopped. This operation takes about 20 seconds.

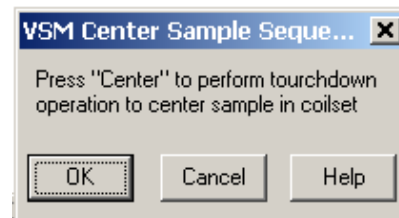


Figure 6-18. Sequence-mode VSM Center Sample command: VSM Center Sample Sequence popup

The **Center Sample** sequence command is invaluable when you have disabled automatic centering (by selecting **No Automatic Centering**), but it is also useful when automatic centering has been enabled. In the latter case, you can place the **Center Sample** command in your sequence just before measurements that you would like to have performed without the interruption of a touchdown operation. The measurement is still subject to the other parameters (e.g., **Averaging Time** and **Logging Interval**) that you have set, but by explicitly executing the **Center Sample** command, you reset the beginning of the interval.

6.8 Sequence-Mode VSM "Moment vs. Field" Command

The sequence-mode **VSM Moment vs. Field** command facilitates using sequences to make common VSM measurements such as hysteresis loops. This command allows you to rapidly set the VSM to take data while sweeping the field in a variety of different patterns, and it gives you the ability to control the spacing of the data points (e.g., uniform spacing in Log (Field)),

1/Field, etc.) so that it is easy to plot the data. When you click on the sequence-mode **Moment vs. Field** command in the **Sequence Commands** bar, it opens the sequence-mode **VSM Moment versus Field** dialog shown in Figure 6-19.

The following describes all of the possible features for this mode. Some features may be irrelevant to your system because of magnet type.

6.8.1 Sequence-Mode VSM "Moment versus Field" Dialog

The **VSM Moment versus Field** dialog has two tabs, **Setup** (Figure 6-19) and **Advanced** (Figure 6-20), with options for setting measurement parameters. As is explained below, the tabs provide several unique functions as well as some of the same functions found in the sequence-mode **Scan Field** and **VSM Adv. Measure** commands.

You can insert the **Moment vs. Field** command at any time by clicking on the **OK** button at the bottom of the dialog. The **Cancel** button closes the dialog and does not insert the command or save any changes you have made to the dialog.

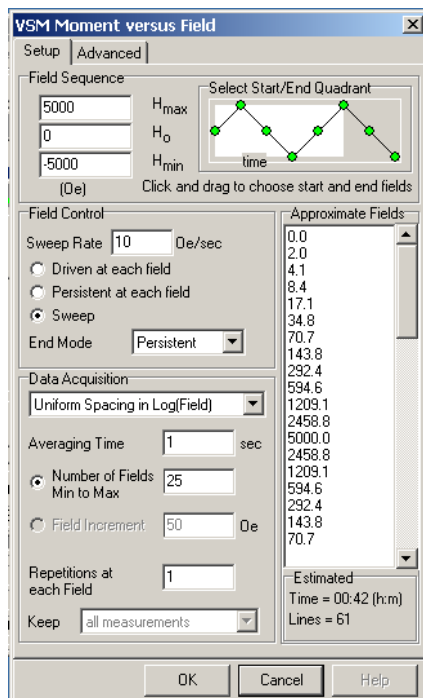


Figure 6-19. Setup tab of the sequence-mode VSM Moment versus Field dialog

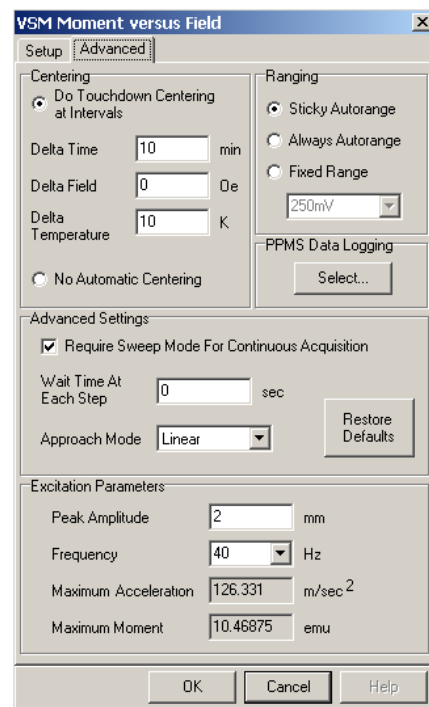


Figure 6-20. Advanced tab of the sequence-mode VSM Moment versus Field dialog

6.8.2 Sequence-Mode VSM "Moment versus Field" Dialog: Setup Tab

The **Setup** tab (Figure 6-19) organizes the parameters relevant to a field-sweep or hysteresis measurement. Options in the **Field Sequence**, **Field Control**, and **Data Acquisition** subsections of the tab allow you to control the settings. A scroll bar at the right side automatically displays **Approximate Fields**. The subsections and their settings are explained below.

6.8.2.1 FIELD SEQUENCE SETTINGS

Use the options in the **Field Sequence** section (Figures 6-21 and 6-22) to set the amplitude and order of the field changes during the **Moment vs. Field** measurement.

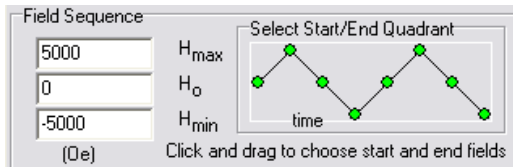


Figure 6-21. Field Sequence section of the VSM Moment vs Field Setup tab: Start-End Quadrant de-selected

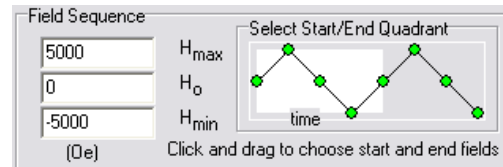


Figure 6-22. Field Sequence section of the VSM Moment vs Field Setup tab: Start-End Quadrant selected

- H_{\max} defines the maximum field used in the measurement.
- H_0 defines a starting or ending field used in the measurement. By default, H_0 is the starting point of the measurement, but you also can use H_{\max} or H_{\min} as the starting point.
- H_{\min} defines the minimum field used in the measurement.

Note: Uniform spacing is referenced between H_{\max} and H_{\min} . This can affect whether H_0 is used in the measurement, as is explained below in the **Data Acquisition** subsection.

- **Select Start/End Quadrant** (Figures 6-21 and 6-22) allows you to use your mouse to set the starting and ending fields of the measurement. To set the starting and ending quadrants (the area between the field set points), place your mouse pointer in the diagram, left-click the field for the start of the measurement, drag the pointer to the end field, and release the left button. The selected quadrants will be highlighted and included in the measurement.

NOTE: Drag the cursor from **left** to **right** when selecting quadrants.

6.8.2.2 FIELD CONTROL SETTINGS

Use the settings in the **Field Control** section (Figure 6-23) of the **Setup** tab to control how the magnetic field changes between the field set points. Some choices will be unavailable due to different magnet types.

Sweep Rate

The **Sweep Rate** value sets the rate at which the field changes when the magnet is ramping up or down.

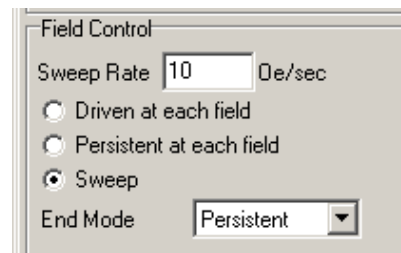


Figure 6-23. Field Control section of the VSM Moment versus Field dialog

Driven at each field

The **Driven at each field** radio button sets the magnet to stabilize the field in **Driven** mode at each field shown in the **Approximate Fields** list (Figures 6-19 and 6-26).

Persistent at each field

The **Persistent at each field** radio button sets the magnet to stabilize the field in **Persistent** mode at each field shown in the **Approximate Fields** list (Figures 6-19 and 6-26).

Sweep

The **Sweep** radio button sets the magnet to continuously change the field without pause during the measurement. When this setting is used, the VSM will collect data at each field shown in the **Approximate Fields** list.

End Mode

The **End Mode** dropdown menu bar allows you to set the state (**Persistent** or **Driven**) that the magnet will be in after the measurement has ended. For more information about **End Mode**.

CAUTION!

For liquid-He based systems, monitor the system closely when it is in **Driven** mode. Use of **Driven** mode significantly increases heat flow into the dewar and the helium boil-off rate, so there is an increased risk of a magnet quench.

6.8.2.3 DATA ACQUISITION SETTINGS

Use the **Data Acquisition** section (Figure 6-24) to set the basic parameters of the moment versus field measurements.

"Data Spacing"

The "**Data Spacing**" dropdown menu bar at the top of the section provides the following options:

- **Continuous Measuring** sets the VSM to take data in **Continuous Measuring** mode. When you are not using **Continuous Measuring**, you must define when the VSM will take data by designating the field increment between measurements, as is explained below in **Number of Fields Min to Max** and **Field Increment**.
- **Uniform Spacing in Field** sets the VSM to uniformly space data collection with respect to the magnitude of the magnetic field.
- **Uniform Spacing in Field²** sets the VSM to uniformly space data collection with respect to the square of the magnitude of the magnetic field.
- **Uniform Spacing in Field^{1/2}** sets the VSM to uniformly space data collection with respect to the square root of the magnitude of the magnetic field.
- **Uniform Spacing in 1/Field** sets the VSM to uniformly space data collection with respect to the inverse of the magnitude of the magnetic field.
- **Uniform Spacing in Log (Field)** sets the VSM to uniformly space data collection with respect to the logarithm of the magnitude of the magnetic field.

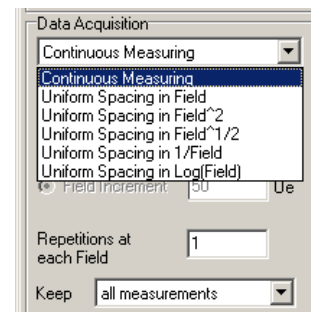


Figure 6-24. Data Acquisition: Data spacing selection

Averaging Time

The **Averaging Time** text box (Figure 6-25) allows you to set the length of time the VSM will collect data before it is averaged into a measurement. See Section 6.4.2.3 for more information about **Averaging Time**.

Number of Fields Min to Max and Field Increment

When you are not using **Continuous Measuring**, you must define the spacing between the fields with one of these options (Figure 6-25). Note that when you select one of these settings, you must enter a number in the associated text box before you will be able to take measurements.

- To set the number of times the VSM will measure between your selected H_{max} and H_{min} , click on the button next to **Number of Fields Min to Max**. The value you enter into the text box next to **Number of Fields Min to Max** sets the number of data points the VSM will take between H_{max} to H_{min} (including H_{max} and H_{min}), spaced uniformly in the manner you have selected.
- To directly set the increment in magnetic field between VSM data points, click on the button next to **Field Increment**. If you select the **Field Increment** radio button, you must enter a number in the associated text box. If you set a step size that does not fit evenly with your selected H_{max} and H_{min} , the program will adjust the increment size slightly to include H_{max} and H_{min} .

Note: Because uniform spacing is referenced between H_{max} and H_{min} , the field you enter for H_0 might not be used in the measurement if the combination of uniform spacing and number of fields does not line up evenly with H_0 . If this occurs, H_0 will only be included as a data point if it is selected as the starting or ending field (or both) of the measurement.

Repetitions at each Field

The value entered for **Repetitions at each Field** (Figure 6-26) is the number of data points the VSM will take at each of the fields shown in the **Approximate Fields** list. This option has no effect if you have selected the **Sweep** or **Continuous Measuring** mode.

Keep

The **Keep** dropdown menu bar (Figure 6-26) is only available when you have selected **Continuous Measurement**, which can generate large volumes of data. Use the **Keep** options to choose a percentage of VSM data points that will be written to the data file. See the **Estimated** explanation below for more on this issue.

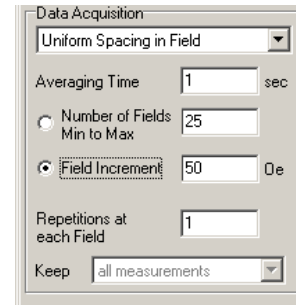


Figure 6-25. Data Acquisition: Number of Fields and Field Increment

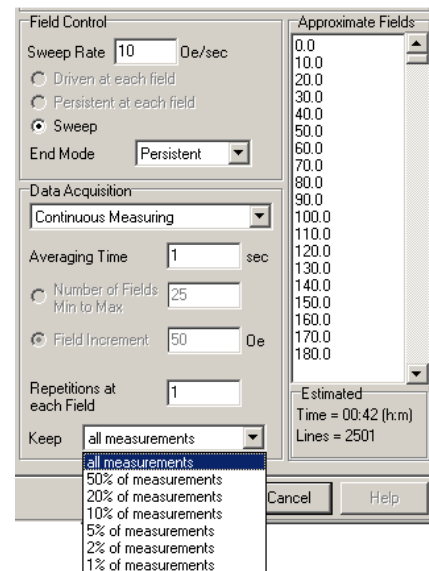


Figure 6-26. Data Acquisition: Keep and Approximate Fields settings

6.8.2.4 APPROXIMATE FIELDS

The **Approximate Fields** list (Figure 6-26) displays, in sequence, a close estimate of the fields where the VSM will take data points, based on all your selections in the **Field Sequence**, **Field Control**, and **Data Acquisition** sections of the **Setup** tab.

6.8.2.5 ESTIMATED

The **Estimated** area (Figure 6-26) displays the estimated amount of time (in hours and minutes) that will be needed to complete the measurement as well as the estimated number of lines in the output data file. The estimated number of lines in the output data file is also the total number of data points generated by your measurement.

6.8.3 Sequence-Mode VSM "Moment versus Field" Dialog: Advanced Tab

The **Advanced** tab (Figure 6-20) organizes the settings for measurement details (e.g., the VSM measurement parameters and the field-approach mode) and has options that control how the VSM takes measurement data. Experienced users will notice that the **Centering**, **Ranging**, **Data Logging**, and **Excitation Parameters** subsections of this tab are identical in form and function to sections of the **VSM Measurement** dialog.

6.8.3.1 CENTERING SETTINGS

Use the **Centering** section of the **Advanced** tab to set the conditions for the VSM to perform touchdown operations. For more information on **Centering**, see Section 6.4.2.4.

6.8.3.2 RANGING SETTINGS

Use the **Ranging** section of the **Advanced** tab to set the way the system chooses the gain of the amplifiers in the VSM module during measurement. For more information on **Ranging**, see Section 6.4.2.5.

6.8.3.3 PPMS DATA LOGGING SETTINGS

The **Select...** button in the **PPMS Data Logging** section opens the **PPMS** dialog (Figure 6-17) so that you can select additional system information to be sent to the output data file. For more information on **Data Logging**, see Section 6.4.2.5.

6.8.3.4 ADVANCED SETTINGS

The **Advanced Settings** subsection (Figure 6-27) contains settings that are rarely needed, but they can provide extra control over your measurements.

Require Sweep Mode for Continuous Acquisition

When you set **Data Acquisition** to **Continuous Measurement** (Section 6.8.2.3), the system automatically uses **Sweep Mode** and the check box next to **Require Sweep Mode For Continuous Acquisition** will be selected.

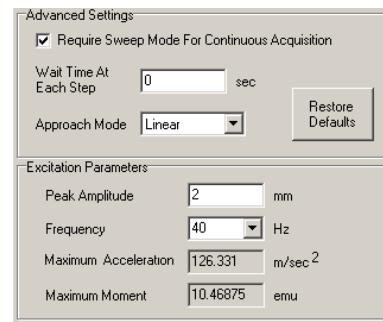


Figure 6-27. Advanced Settings—Advanced tab: Require sweep, Wait Time, and Approach Mode settings

Although you can clear the check box and select the **Driven at each field** or **Persistent at each field** mode during continuous measurements, be aware that these modes might increase the noise and introduce artifacts into your data.

Note: If you measure continuously while the power supply stabilizes the magnetic field at each set point, the noise level might increase temporarily, introducing artifacts into the VSM data until the system can achieve field stability.

Wait Time At Each Step

The **Wait Time At Each Step** text box is used to set the *additional* time (in seconds) that you want the system to wait at each field set point. This wait time is in addition to the amount of time it takes for the required VSM measurements and it is added *before* the VSM performs the measurements. The **Wait Time At Each Step** setting has no effect if you have selected **Sweep** or **Continuous Measuring** modes.

Approach Mode

The **Approach Mode** dropdown menu bar lets you choose between **Linear**, **No O'Shoot** (No Overshoot), and **Oscillate** field-approach modes for each of the magnetic field set points in your measurement. We recommend **Linear** mode when your **Data Acquisition** setting is **Continuous Measuring**.

Restore Defaults

The **Restore Defaults** button resets everything in the dialog to the default settings.

6.8.3.5 EXCITATION PARAMETERS

Use the options in the **Excitation Parameters** subsection (Figure 6-27) to set the drive variables for the VSM linear motor transport. For more information on these options, see Section 6.4.2.5.

6.9 Sequence Mode VSM "Moment vs. Temp." Command

The VSM sequence-mode **Moment vs. Temp.** command is designed to make these types of measurements easy to set up and execute in a sequence. You can use the **Moment vs. Temp.** command to rapidly set the VSM to take data while sweeping temperature in a variety of different patterns. You also can control when data is taken with respect to temperature increment (e.g., uniform spacing in **Log (Field)** or **1/Field**), which facilitates plotting the data. When you click on the sequence-mode **Moment vs. Temp.** command in the **Sequence Commands** bar, the sequence-mode **VSM Moment versus Temperature** dialog opens (Figures 6-28 and 6-29).

6.9.1 Sequence-Mode VSM "Moment versus Temperature" Dialog

The **VSM Moment versus Temperature** dialog has two tabs, **Setup** (Figure 6-28) and **Advanced** (Figure 6-29). Use the **Setup** tab to set the parameters for a variable temperature measurement. Use the **Advanced** tab to control other aspects of the measurement, such as the temperature-approach mode.

As is explained below, the tabs have several unique functions as well as some of the same functions found in the sequence-mode **Scan Temperature** and **VSM Adv. Measure** commands.

You can insert the **Moment vs. Temp.** command at any time by clicking on the **OK** button at the bottom of the dialog box. The **Cancel** button closes the dialog and does not insert the command.

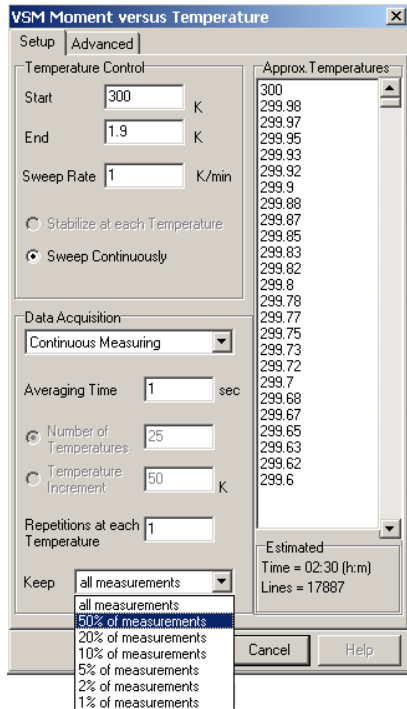


Figure 6-28. Setup tab of the sequence-mode VSM Moment versus Temperature dialog

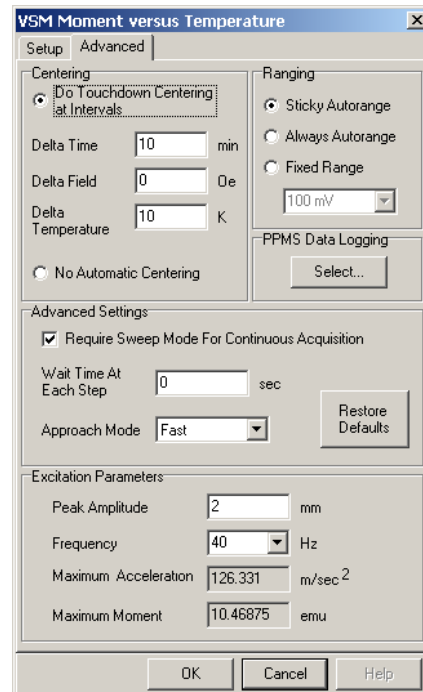


Figure 6-29. Advanced tab of the sequence-mode VSM Moment versus Temperature dialog

6.9.2 Sequence-Mode VSM "Moment versus Temperature" Dialog: Setup Tab

The **Setup** tab (Figure 6-28) has subsections for **Temperature Control** and **Data Acquisition**. A scroll bar at the right side automatically displays **Approx. Temperatures** that are based on the other settings. The subsections and their settings are explained below.

6.9.2.1 TEMPERATURE CONTROL SETTINGS

Use the options in the **Temperature Control** subsection (Figure 6-30) of the **Moment versus Temperature** dialog box to set the general parameters of a moment versus temperature measurement.

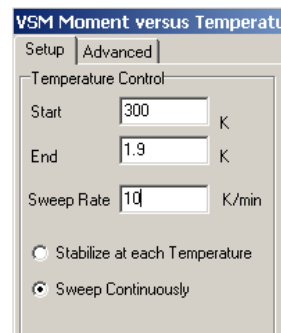


Figure 6-30. Temperature Control section of the sequence-mode VSM Moment versus Temperature dialog

Start and End

The **Start** value sets the temperature, in degrees Kelvin, at which the measurement will start during a temperature sweep. The initial temperature can be larger or smaller than the final temperature, so you can collect data while the sample is being warmed or cooled.

The **End** value sets the temperature, in degrees Kelvin, at which the moment versus temperature measurement will end.

Sweep Rate

The **Sweep Rate** value sets the rate at which the temperature changes, in degrees Kelvin per minute, during a measurement. The **Moment vs. Temp.** sequence command interprets the **Sweep Rate** as a magnitude and uses the **Start** and **End** temperatures to determine the sign of the temperature-sweep rate.

Note: If you choose to stabilize the temperature during measurements, the **Sweep Rate** will become the rate of temperature change between temperature set points instead of the overall rate of temperature change.

Stabilize at each Temperature and Sweep Continuously

The **Stabilize at each Temperature** and **Sweep Continuously** options provide two modes for sweeping temperature. When you select **Stabilize at each Temperature**, the system waits for the temperature to stabilize at each of the set points listed in the **Approx. Temperatures** window before it begins to take data. When you select **Sweep Continuously**, the system changes temperature at the **Sweep Rate** setting while the VSM takes data, and it does not pause at the set points listed in the **Approx. Temperatures** window.

6.9.2.2 DATA ACQUISITION SETTINGS

Use the options in the **Data Acquisition** subsection of the dialog box to set the basic parameters of the moment versus temperature measurement.

"Data Spacing"

The "Data Spacing" dropdown menu bar at the top of the section provides the following options:

- **Continuous Measuring** (Figures 6-28 and 6-32) sets the VSM to take data in **Continuous Measuring** mode (refer to Section 6.2.4.3 for more information on **Continuous Measuring**). When you are not using **Continuous Measuring**, you must define the temperature change between measurements, as is explained below in **Number of Temperatures** and **Temperature Increment**.
- **Uniform Spacing in Temperature** (Figure 6-31) sets the VSM to take data at uniform temperature intervals.
- **Uniform Spacing in Temperature²** sets the VSM to take data at uniform temperature intervals with respect to the square of the temperature.
- **Uniform Spacing in Temperature^{1/2}** sets the VSM to take data at uniform temperature intervals with respect to the square root of the temperature.

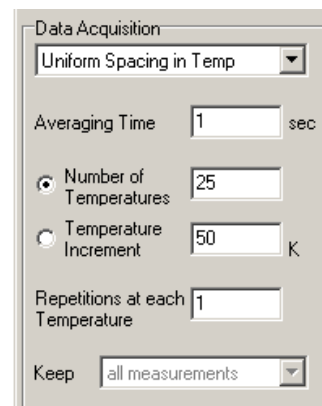


Figure 6-31. Data Acquisition section of the sequence-mode VSM Moment versus Temperature dialog

- **Uniform Spacing in 1/ Temperature** sets the VSM to take data at uniform temperature intervals with respect to the inverse of the temperature.
- **Uniform Spacing in Log (Temperature)** sets the VSM to take data at uniform temperature intervals with respect to the logarithm of the temperature.

Averaging Time

The **Averaging Time** value (Figures 6-31 and 6-32) sets the length of time the VSM collects data before it is averaged into a measurement. Section 6.4.2.3 has more information about **Averaging Time**.

Number of Temperatures and Temperature Increment

When you select a uniform temperature spacing instead of **Continuous Measuring**, you must define the spacing between the temperatures with the **Number of Temperatures** or **Temperature Increment** options (Figure 6-31).

- The **Number of Temperatures** value sets the number of temperature set points at which VSM data will be taken. This number of temperature set points will be distributed uniformly over your defined temperature range in the manner you have selected. If you select the **Number of Temperatures** radio button, you must enter a number in the associated text box.
- The **Temperature Increment** value sets the temperature increment, in degrees Kelvin, between VSM measurements. If you select the **Temperature Increment** radio button, you must enter a number in the associated text box.

Note: If you set a step size that does not fit evenly with your selected **Start** and **End** temperatures, the program will adjust the increment size slightly to include the **Start** and **End** temperatures.

Repetitions at each Temperature

The **Repetitions at each Temperature** value (Figure 6-32) sets the number of data points the VSM will take at each of the temperatures shown in the **Approx. Temperatures** list. This value will have no effect if you have also selected the **Sweep** or **Continuous Measuring** modes.

Keep

The **Keep** dropdown menu bar (see Figure 6-32) is available when you have selected the **Continuous Measurement** option, which can generate large volumes of data. Use the **Keep** options to choose the percentage of data points that will be written to the data file. See the **Estimated** explanation below for more on the issue of file size.

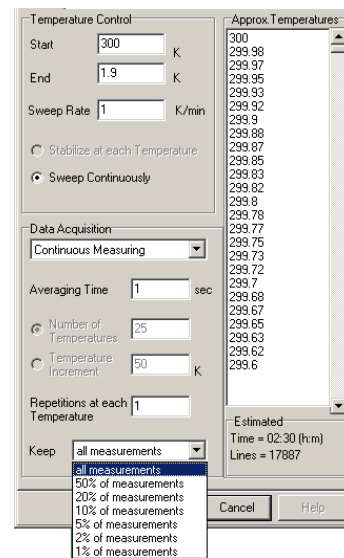


Figure 6-32. Data Acquisition section: Keep, Approximate Temperature, and Estimated settings

6.9.2.3 APPROX. TEMPERATURES

The **Approx. Temperatures** list (Figure 6-32) displays, in sequence, a close estimate of the temperatures at which the VSM will take data. These temperatures are based on all your selections in the **Temperature Control** and **Data Acquisition** sections of the **Setup** tab.

6.9.2.4 ESTIMATED

The **Estimated** area (Figure 6-32) displays the estimated amount of time (in hours and minutes) that will be needed to complete the measurement as well as the estimated number of lines in the output data file. The number of lines in the output data file is also the total number of data points that will be generated by your measurement. For instance, there will be 17887 lines of data in the example measurement in Figure 6-32.

6.9.3 Sequence-Mode VSM "Moment versus Temperature" Dialog: Advanced Tab

The options in the **Advanced** tab (Figure 6-29) of the sequence-mode **VSM Moment versus Temperature** dialog control how the VSM takes data during a measurement. Experienced users will notice that many sections of this tab are identical in form and function to sections of the immediate-mode **VSM Measurement** dialog.

6.9.3.1 CENTERING SETTINGS

Use the **Centering** subsection of the **Advanced** tab to set the conditions for the VSM to perform touchdown operations. For more information on **Centering**, see Section 6.4.2.4.

6.9.3.2 RANGING SETTINGS

Use the **Ranging** subsection of the **Advanced** tab to set the way the system chooses the gain of the amplifiers in the VSM module during measurement. For more information on **Ranging**, see Section 6.4.2.5.

6.9.3.3 PPMS DATA LOGGING SETTINGS

The **Select...** button in the **PPMS Data Logging** subsection opens the **PPMS** dialog (Figure 6-17), which provides additional system information that you can have sent to the output data file. For more information on **PPMS Data Logging**, see Section 6.4.2.5.

6.9.3.4 ADVANCED SETTINGS

The **Advanced Settings** subsection (Figure 6-33) is a general section for controls that are rarely used.

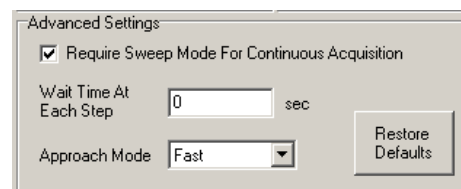


Figure 6-33. Advanced Settings section: Require sweep, Wait Time, and Approach Mode

Require Sweep Mode For Continuous Acquisition

The system will automatically activate the **Require Sweep Mode For Continuous Acquisition** option when **Continuous Measurement** data acquisition has been selected.

Note: You can uncheck the box and use the **Stabilize at each Temperature** mode while you perform continuous measurements. However, be aware that if you measure continuously while the controller is stabilizing the temperature, the amount of noise in the VSM data might increase.

Wait Time At Each Step

The **Wait Time At Each Step** value sets the additional time (in seconds) that the system waits at each of the temperature set points. This wait time is in addition to the amount of time it takes to do the required VSM measurements; it is added *before* the VSM performs the measurements. This setting has no effect if you have selected **Sweep** or **Continuous Measuring** modes.

Approach Mode

The **Approach Mode** dropdown menu bar lets you choose between **Fast** and **No O'Shoot** temperature-approach modes for each of the temperature set points in your measurement.

Restore Defaults

The **Restore Defaults** button resets everything in the dialog to the default settings.

6.9.3.5 EXCITATION PARAMETERS

Use the options in the **Excitation Parameters** subsection to set the drive variables for the VSM linear motor transport. For more information on **Excitation Parameters**, see Section 6.4.2.5.

6.10 VSM Data Files

Data files have a `.dat` file extension. To save VSM measurement data, you must open a measurement data file *before* you start the measurement. You designate data files by creating a new one or selecting a pre-existing one, as is explained in Section 6.4.1.3.

6.10.1 Data File Headers

The header of a data file contains information such as the title of the data set and the sample properties. You have the opportunity to include this information when you create the file—this is the only time you can add this information to the data-file header. Instead, after a file has been created, you can append comments.

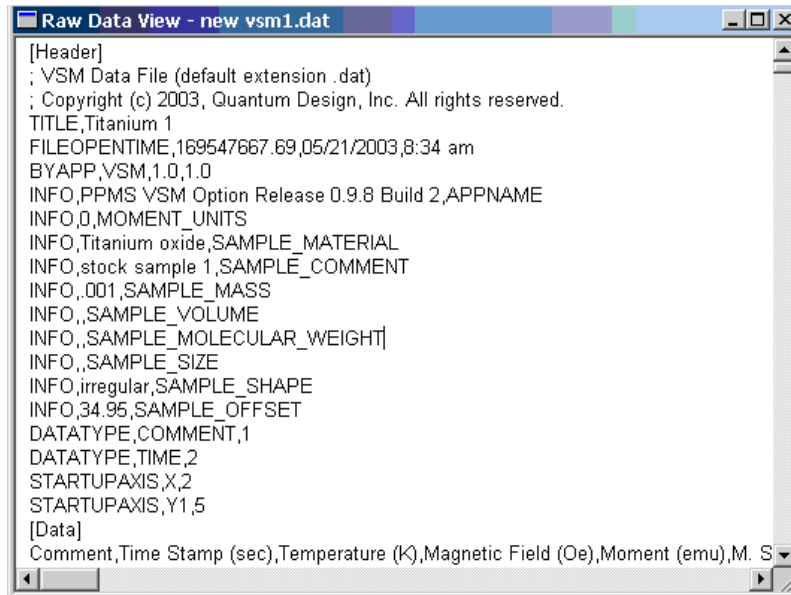


Figure 6-34. A Raw Data view of a VSM data file

6.10.2 Fields in VSM Data Files

Table 6-1 defines the fields in a VSM data file, and Section 6.4.1.3 explains how to create VSM measurement data files.

Table 6-1. Definitions of column headers for VSM data files (*.dat files), shown in the order they appear.

COLUMN HEADER/TERM	DEFINITION
Comment	user-specified comment; added by using the Datafile Comment command
Time stamp (sec)	time stamp
Temperature (K)	average temperature (T) of the sample during measurements. The sample temperature is measured by the coil thermometer.
Magnetic field (Oe)	average magnetic field during measurement
Moment (uu)	average magnetic moment of the sample during measurement; uu = user units = emu or A-m ²
M. std err (uu)	standard error (i.e., the error of the mean) for the measurement
Transport action	1 = measurement 2 = auto-touchdown 3 = manual touchdown
Averaging time (sec)	(number of cycles per measurement)/frequency (as calculated)
Frequency (Hz)	frequency of sample oscillation
Peak amplitude (mm)	peak amplitude (A) of oscillation, such that position $z(t) = A \sin \omega t$
Center position (mm)	Average position of the transport for the current data point. Position is reported in motor coordinates and does not reflect the sample offset position that is shown in the locate dialog.
Coil signal' (mV)	uses known phasors for preamp and board to back out the actual voltage signal registered at the coilset in phase with the sample motion; does not correct for the image effect
Coil signal'' (mV)	quadrature component of above quantity
Range (mV)	VSM board range setting; this can be 0.25, 2.5, 25 or 250 mV

COLUMN HEADER/TERM	DEFINITION
M. quad. signal (uu)	lock-in quadrature component of "Moment (uu)" field
M. raw' (emu)	moment with no temperature dependent correction (image effect or coil shrinkage) applied
M. raw'' (emu)	quadrature component of above quantity
Min. temperature (K)	minimum temperature reading of the coil thermometer for this measurement
Max. temperature (K)	maximum temperature reading of the coil thermometer for this measurement
Min. field (Oe)	minimum field reading of the coil thermometer for this measurement
Max. field (Oe)	maximum field reading of the coil thermometer for this measurement
Mass (grams)	mass of transport as obtained from DC component of motor force
Motor lag (deg)	phase lag between motor drive current and motion
Pressure (torr)	pressure in sample chamber
VSM status (code)	status codes unique to the VSM module
Motor status (code)	status codes unique to the motor module
Measure status (code)	error condition codes of varying severity. Zero indicates no errors.
Status (code)	status code for base system
System temp. (K)	block temperature
System field (Oe)	currently the same as the "Magnetic Field (Oe)" column
Pressure()	pressure in sample chamber as reported by high vacuum gauge (when present)
Map 25()	VSM coil set temperature

Troubleshooting

7.1 Introduction

This chapter contains the following information:

- Section 7.2 provides an overview of the Troubleshooting chapter.
- Section 7.3 discusses possible results of a vibrating coil.
- Section 7.4 outlines how the sample center changes with temperature and its importance in temperature-dependant magnetic measurements.
- Section 7.5 discusses the effects of a loosely mounted sample on measurement results.
- Section 7.6 discusses how high fields (14T and above) can affect magnetic measurements.

7.2 Overview

This chapter discusses some issues – artifacts or noise – that can arise when making VSM measurements and describes ways of mitigating or preventing the issues. Most of the material in this chapter is taken from VSM application notes that are posted on the Quantum Design website www.qdusa.com and have thus been incorporated into this user manual.

It is useful to classify the measurement artifacts into two classes: 1) those that affect the *accuracy* of the reported moment, and 2) those that affect the *precision* (i.e., the noise level). While poor precision will often be easy to recognize (high values of M.Std.Err. and noise in the Moment value), it is more challenging to determine if the moment is being reported accurately.

Below are some important topics to consider when troubleshooting VSM data. To help you decide the relevance of each to your particular data, the section starts with a list of common symptoms associated with this issue.

7.3 Vibration of the Detection Coil Set

Symptoms:

- *noisy moment at high fields*
- *features in $M(H)$ like unexpected curvature or shifts*
- *heating at low temperatures which occurs only when sample is oscillating*

In a VSM it is important that the detection coil set is immobile because any vibrations of it relative to the magnetic field will create induced voltages. A coil with many windings and which is located in a very large (and slightly non-uniform) magnetic field will be a very sensitive microphone! The VSM sample rod is guided by special blue plastic alignment bearings in the sample chamber. There is necessarily some friction as the sample rod oscillates and this can transmit vibrations into the coil set. In addition, direct rubbing of the sample in the coil set can occur if the sample rod/sample holder assembly is not straight or if the sample is large diameter and contacts the inner bore of the coil set directly. These induced vibrations are of the same frequency as those due to the moving sample and so are indistinguishable from sample signal by the lock-in amplifier. Fortunately, this signal statistically appears equally in both the Moment and M.Quad.Signal (quadrature “moment”, or 90-degree phase lagged in time) values. Since this quadrature portion is not due to the sample’s magnetic moment (to a precision of <0.5%), signal here will be a very helpful clue in understanding the magnitude of the parasitic signal in the reported moment.

Methods of mitigating these unwanted coil set vibrations are outlined below.

- 1) Ensure the sample rod and sample holder are straight by viewing down the length and rotating the rod. Excessive “wobble” will lead to increased chances of contact with the coils. Note that perfect straightness is not possible to achieve.
- 2) Ensure the sample diameter is less than (coil set bore) – 2mm. The standard coils have a 6mm bore while the large bore coil set is 12mm. It is helpful to have a large bore coil set (P529) on-hand in order to rule out such frictional issues.
- 3) Cut the vibration frequency in half (typically from 40 Hz to 20 Hz) and remeasure. As the accelerations and forces scale with the square of the vibration frequency, this means a 75% decrease in forces and may in some cases provide a dramatic improvement. Note that the VSM hardware is calibrated only near 40 Hz so it is important to check the calibration using the Pd standard sample if a new frequency is chosen. To change the vibration frequency for a one-time test, this can be simply typed into the box in the Advanced tab of the VSM measure dialog. To change the dropdown menu of available frequencies in this dialog, consult PPMS service note 1096-304.
- 4) Make sure that an Extended Purge was performed at the end of the Sample Install wizard if measurements are being made at low temperatures. This will prevent the formation of ice on the bearing surfaces. A test for roughness in the bearings is performing the Motor Friction Scan on the Advanced tab of the VSM Control Center. Typical hysteresis with a sample rod inserted is 10-20 mA.

7.4 Sample Centering and Temperature-Dependent Magnetization Measurements

(VSM Application Note 1096-305)

Symptoms:

- *inaccurate moment at low temperatures*
- *steps in $M(T)$*
- *low temperature $M(H)$ hysteresis loops that do not close*

When making VSM measurements in the PPMS family of instruments (PPMS, DynaCool or VersaLab), the sample is positioned in the detection coil set by suspending it from a carbon fiber sample rod which is held in the VSM motor. The coil set is plugged into the sample chamber, which in the PPMS and DynaCool is an 87 cm long sealed thin-walled metal tube of which the upper 75 cm are type 304 stainless steel and the lower 12 cm are high purity copper. In the case of the shorter VersaLab chamber, the upper stainless section is only 17 cm long. This shorter chamber as well as the reduced temperature range (50-400 K) in VersaLab will reduce the effects discussed here, thus this application note will focus on the PPMS and DynaCool platforms.

The VSM motor position is fixed at the top of the sample chamber¹. Thus, the centering of the VSM sample will be affected by relative length changes in the sample chamber versus the VSM sample rod. The carbon fiber of the sample rod has a very low thermal expansion coefficient, while the stainless steel of the (PPMS and DynaCool) sample chamber will contract in length by ~2mm when the sample region temperature changes from 300 K to 2 K. The VSM software provides a sample centering mechanism which is referred to as the “touchdown” operation, and compensates for this relative shift by periodically touching the end of the sample holder down to the VSM detection coil set base so that the current position of the sample relative to the coil set is determined (see the relevant *VSM Option User Manual* for your platform for more details on this).

Figure 7-1 shows a VSM data file for a moment vs. temperature sweep in which touchdown sample centering was performed every 10 K or 10 minutes (these are the default settings). The “Center Position (mm)” quantity refers to the motor’s position where 65 mm is the top of travel and 0 mm is the bottom, thus any changes to this value will reflect relative length changes between the sample rod and sample chamber. An increase in the center position value means that the sample chamber has contracted relative to the sample rod. This temperature scan was done very fast (~ - 7.5 K/min) in order to demonstrate the effect of thermal contraction – note the logarithmic axis for temperature.

The main point in this figure is that the sample position is moving for **4 hours after temperature is stabilized at the VSM coil set**. This is because of the large thermal mass of the middle portion of the stainless steel tube and the fact that the temperature control algorithm is optimized in order to stabilize the temperature at the sample location, not in the middle of the sample chamber. In other words, it takes several hours for the thermal profile of the sample chamber to stabilize after a new sample temperature is established. In this case, the chamber appears to be expanding slightly over these 4 hours, which could be due to the fact that the very fast cool down under-cooled the middle part of the sample chamber.

¹ The one exception to this is on the 16 tesla PPMS system in which the large magnetic field gradients cause the motor to be pulled down slightly at high magnetic fields, necessitating sample centering as a function of magnetic field.

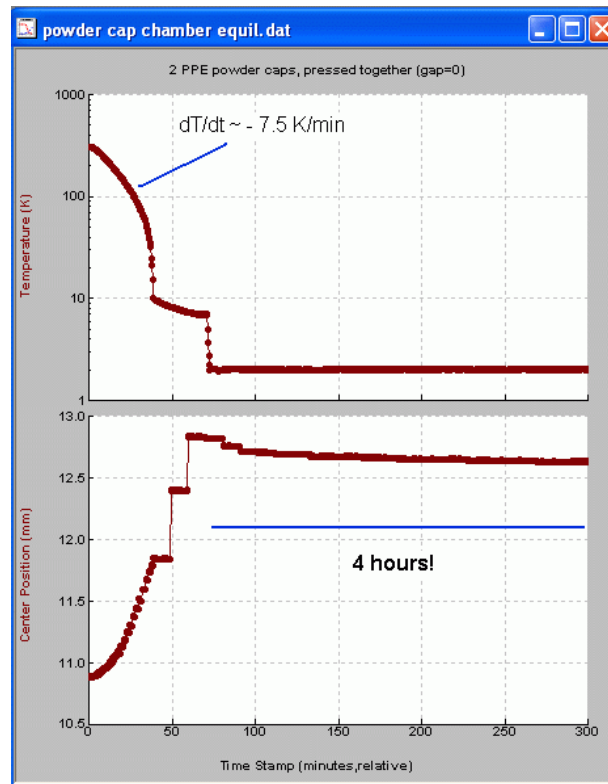


Figure 7-1. VSM data file showing change in sample position as temperature is lowered.
Note: Log temperature axis.

The reason that sample centering is so important is that the reported magnetic moment depends on the vertical position of the sample relative the center of the coils. To understand how an error in vertical centering translates into an error in the reported moment, one must perform a centering scan on the sample as is typically done in the sample installation wizard of the VSM software. This data for the most recent scan is stored in the file `ScanData.dat` located in the `\VSM\LogFiles` folder. Figure 7-2 shows centering scans for the sample measured here (left) as well as an ideal sample (right). The vertical axis *Source*² corresponds to the magnetic moment in units of emu while the horizontal axis *Position* is the motor position in units of mm. The sample used for this investigation was two empty VSM powder sample holders pressed together, and was chosen because the reported moment was a strong function of position (change in reported moment vs. vertical position $dM/dz \sim 20\%$ per mm). When measuring small magnetic moments, this often occurs because the magnetic “end effect” of the sample holder or other nearby material (like the powder capsules) can dominate over the magnetic response due to the sample. In contrast, the ideal sample exhibits an extremum at the sample location so that the moment is independent of position to a first order approximation.

² in older versions of VSM software, one must plot *M.R.* instead of *Source* on the vertical axis.

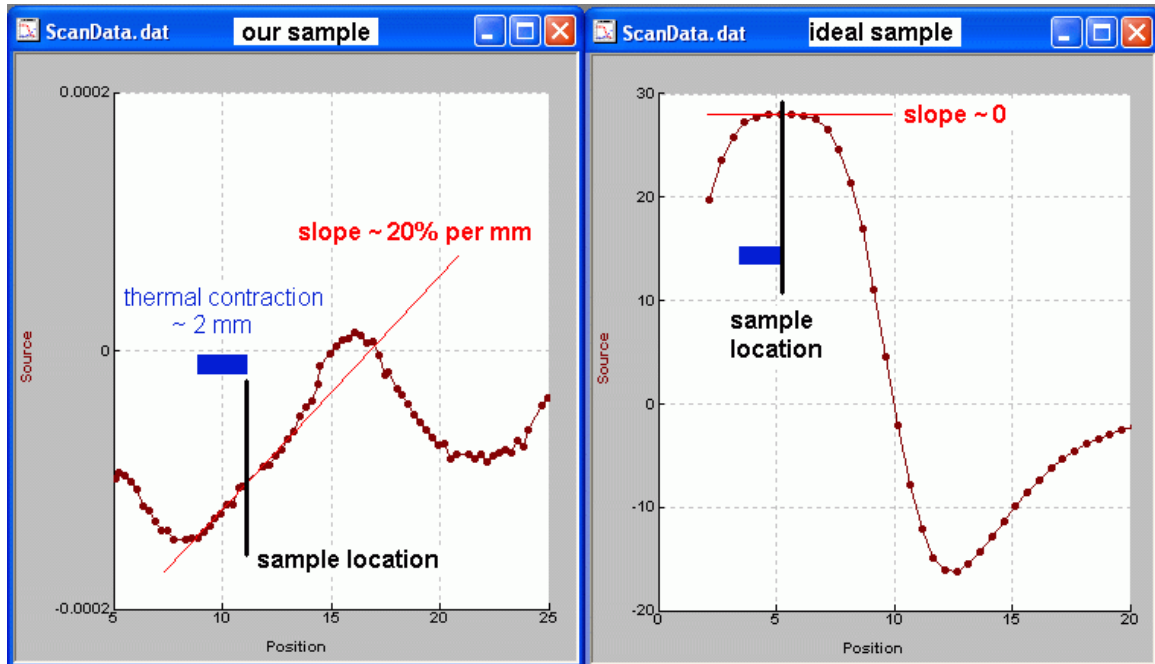


Figure 7-2. Centering scan data taken in 1 tesla applied field for the sample used in this investigation (left) as well as an ideal sample (right). Blue bar shows position shift expected when cooling from 300 K to 2 K without performing touchdowns.

An example of the artifact in the reported moment is shown in Figure 7-3, where the sample thermometer has reached a plateau near 10 K during cooling and hence the touchdown centering operations occur less frequently. That is, they occur only at 10 min. intervals, instead of the ~1.5 min. intervals which corresponded to 10 K changes at higher temperatures. However, it can be seen from the Center Position vs. time plot that **the sample chamber is still contracting at the same rate despite the fact that the VSM sample thermometer has reached a stable temperature**. This means that the sample walks off by as much as 0.6mm over the course of 10 minutes, and the reported moment drifts accordingly.

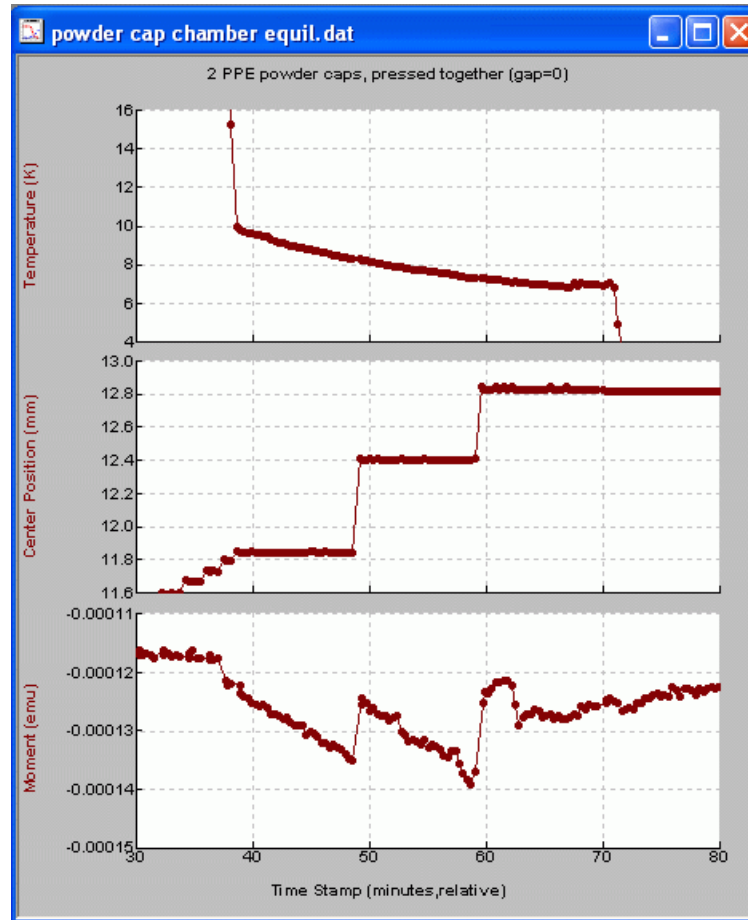


Figure 7-3. Showing artifact in reported moment due to errors in vertical centering.

Thus, the following points should be kept in mind whenever making VSM measurements, and especially when temperature-dependent measurements are being performed:

- 1) When making temperature scans, touchdown every 10 K.
- 2) When at stable temperature, touchdown every 10 minutes unless you are confident that the sample chamber thermal profile is stable. Keep in mind that this will be affected by any temperature changes within the last 4 hours.
- 3) Try to mount your sample so that the reported moment is not a strong function of vertical position (see “ideal sample” centering scan in Figure 7-2).

If the touchdown centering operation is not appropriate to your sample, it is possible to activate an advanced centering option which allows you *within a measurement sequence* to re-center the sample by scanning or by setting an absolute motor position. Keep in mind that there are more risks involved when automating the centering scan or when setting the absolute motor position that can result in damage to the sample, sample rod, or even the motor. Please contact apps@qdusa.com for more information if you would like to access this advanced centering feature.

7.5 Mounting Samples Loosely Causes Moment Noise

(VSM Application Note 1096-303)

Symptoms:

- noise in both *Moment* and *M.Quad Signal*
- noise level scales with the magnitude of the moment
- noise that abruptly starts or stops as a function of temperature

Precise VSM measurements require smooth sinusoidal motion of the sample within the pickup coils. Deviations from this ideal motion will produce artifacts in the measured magnetic moment of the sample. This can occur in the following cases:

- A sample is not held tightly in the sample holder and subsequently it rattles
- A powdered sample is not packed tightly and the material shakes within the sample holder
- A glue joint on the sample rod is loose so that the sample holder slips.

Figure 7-4 shows magnetic moment vs. temperature data from a nickel sphere 2 mm in diameter, measured in a magnetic field of 1 tesla. The moment is very large (~3.5 emu) and at temperatures below about 290 K its behavior is noisy and erratic, based on the following observations:

The moment data exhibits significant noise and jumps.

The quadrature signal “M Quad. Signal”, which describes any coilset pickup that is not in phase with the motion of the motor, is a significant fraction (~5%) of the moment signal.

The standard error “M. Std. Err.,” which describes the uncertainty in the reported moment value, is scattered.

In contrast, at temperatures above 300 K the moment data is smooth, and the quadrature signal and standard error are both less than 0.5% of the sample moment.

The noisy data is explained by the fact that the sample was not moving sinusoidally with the motor, but was instead rattling around in the sample holder. A significant quadrature component arose because the loose sample lagged behind the motion of the motor.

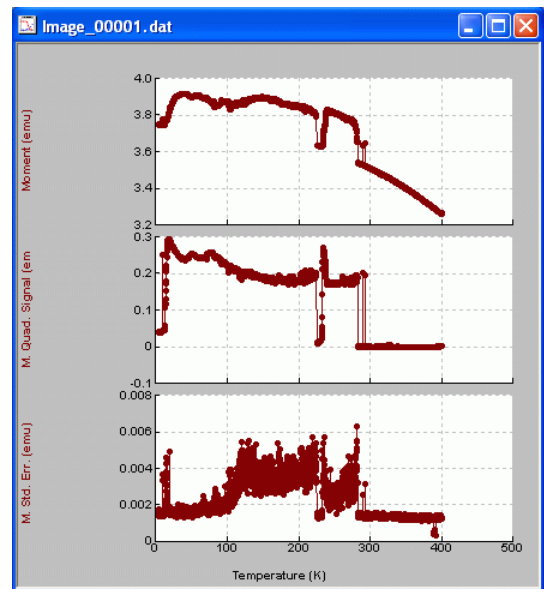


Figure 7-4. Magnetization vs. temperature data for nickel, with sample rattling in the sample holder below 290 K

Differential thermal contraction of materials is a common cause of loose joints between parts, and it could explain why the noise in this data only appeared at temperatures below 290 K.

Figure 7-5 is a photograph of an oscilloscope trace of the signal for the same sample used above. The signal was taken from the BNC monitor (“MONITOR JB-2”), which is located on the front panel of the VSM CM-B module, during the measurement. Note the jagged distortion of the signal, which appears strongest during a particular part of the cycle.



Figure 7-5. Oscilloscope trace of 40 Hz coilset signal, measured at the BNC on the VSM module. Note the jagged distortion of the signal, which would be smooth and sinusoidal for a well-mounted sample.

The following suggestions should help prevent problems due to sample mounting.

- 1) Verify that the sample is rigidly mounted in the sample holder, keeping in mind that the accelerations during VSM measurements are very high!
Noting that $\text{acceleration} = (\text{amplitude}) * (\text{frequency})^2$, then for a VSM peak amplitude of 2 mm and a frequency of 40 Hz, the acceleration is as high as 126 m/sec^2 during a cycle.
- 2) Use an adhesive to hold the sample, if possible. Examples of recommended low-temperature adhesives are GE 7031 varnish or Devcon Duco cement. These are both soluble in common laboratory solvents: a toluene:alcohol mixture in the case of the GE varnish, and acetone in the case of the Duco cement.
- 3) Inspect all glue joints on the sample rod and sample holder for loose connections.
- 4) Verify that powdered samples are packed tightly so that they are immobilized.

7.6 Performing VSM Measurements in PPMS High-Field (14 T or Higher) Magnets

(VSM Application Note 1096-301)

Symptoms:

- *noise in Moment when measuring during a field sweep at low fields*

The VSM option for the PPMS offers rapid measurements—data can be collected fast (greater than 1 Hz data rate) and it can be collected while the magnetic field is being ramped. However, when a 14 T (or higher) magnet is used to measure between -2 T and $+2 \text{ T}$, Quantum Design advises users to measure at stable magnetic fields. The reason for this advice is that there is

significant magnetic noise emanating from the superconducting magnet at these fields, and this is picked up by the VSM detection coilset, which lies in the center of the magnet.

The noise originates from *magnetic flux jumps* in the windings of the high-field superconductor insert, which is composed of Nb₃Sn wire. Magnetic field penetrates a superconductor in the form of miniscule discrete flux lines. The magnet windings immobilize (or pin) the magnetic flux lines, and Nb₃Sn wire is especially good at this. A trade-off of such strong pinning is that, when the field density in the magnet bore is sufficiently different than the pinned flux in the windings, the flux lines in the superconductor tend to be released and trapped rapidly. The field equilibrates during such flux-jump events as magnetic field enters or leaves the windings in small avalanches of flux lines. This equilibration tends to occur when the field-charging direction is reversed at low field magnitudes (less than 2 T) or when the sign of the field changes. Flux jumping subsides at higher fields because the magnet is "saturated" with flux lines.

Another aspect of high flux pinning in the magnet is large remanent magnetic fields in the sample space (about 100 Oe for the 14-T magnets used currently at Quantum Design, which are fabricated using the "internal tin process" Nb₃Sn magnets). Remanence is the field that remains in the magnet after it has been brought back to zero current from full field.

Flux jumping is not a problem in the low-field magnets (up to 9 T), because the windings are made of NbTi superconducting wire, which does not pin flux as strongly as the Nb₃Sn wire and which has not been found to present a problem for VSM measurements while ramping the field. Additionally, the remanence in these magnets is generally less than about 10 Oe at the sample location.

Figure 7-6 illustrates flux jumps and their effects on moment versus field data collected with a 14-T magnet during ramping and stable fields. The data show flux-jump noise in the magnetic moment while the field is ramping, but the noise has settled by the beginning of the field-iteration phase. Thus, by the time that field stability has been declared (labeled "Holding" in Figure 1), the flux jumps have clearly subsided. Note that the driven mode was used for the measurements presented in Figure 7-6.

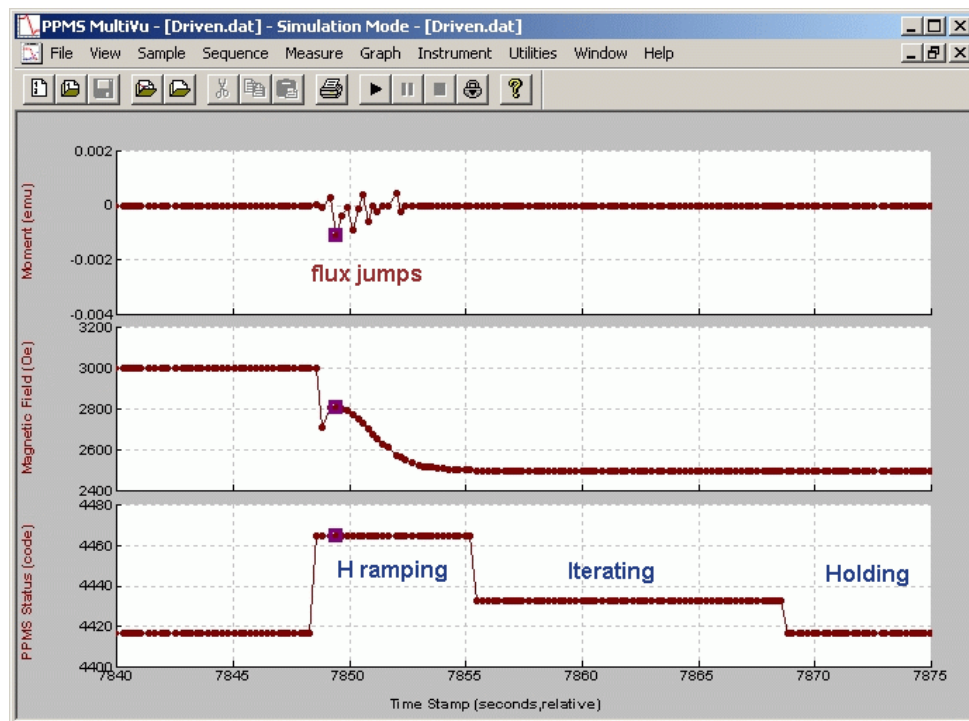


Figure 7-6. The effect of flux jumps on VSM moment versus field data during ramping and stable fields when using a 14-T magnet at fields between -2 T to +2 T.

Thus, Quantum Design strongly advises users to measure moment versus field in a stepwise manner and perform measurements only at stable fields when measuring between -2 T to $+2$ T. In the event that a user would like to sweep the field over the full range in a 14-T magnet, below is a model for constructing a sequence file that helps minimize flux jumps.

SEQUENCE MODEL: M(H) FROM +14 TESLA TO -14 TESLA

VSM M(H) command: +14 tesla to +2 tesla, 100 Oe/sec, SWEEP mode, end mode = driven, measure continuous

VSM M(H) command: +2 tesla to -2 tesla, 100 Oe/sec, DRIVEN AT EACH FIELD mode, end mode = driven, Uniform spacing in field

VSM M(H) command: -2 tesla to -14 tesla, 100 Oe/sec, SWEEP mode, end mode = driven, measure continuous

Set Field 0.0, end mode = persistent

Model CM-A VSM Motor Module

A.1 Introduction

This appendix contains the following information:

- Section A.2 provides a functional overview of the Model CM-A VSM motor module, including a block diagram and electrical specifications.
- Section A.3 describes the front panel and relevant components of the Model CM-A VSM motor module.
- Section A.4 describes the back panel and relevant components of the Model CM-A VSM motor module.
- Section A.5 describes maintenance of the Model 1000.

A.2 Functional Overview

The Model CM-A (4101-100) is a servomotor controller module that was designed with the specific needs of the VSM head in mind. Figure A-1 shows the module and the front panel.

The principle function of this module is to provide closed-loop servo control to a linear motor equipped with a position encoder output. A programmed wave table allows the module to drive the motor sinusoidally at 40 Hz. The servo loop is closed digitally at about 2000 Hz using a 16-bit current source and the read-back from the position encoder. For use with other synchronous detection hardware, including the Model CM-B VSM detection module, the real-time encoder position is output digitally, using a high-speed serial port, and as a voltage through a BNC connector. Other features include in-system programmable on-board flash memory for program storage and a serial ROM for calibration and other configuration data.

The module is designed to plug into the Model 1000 modular control system or an equivalent host chassis that can provide power and the required CAN network signals that communicate with the module.

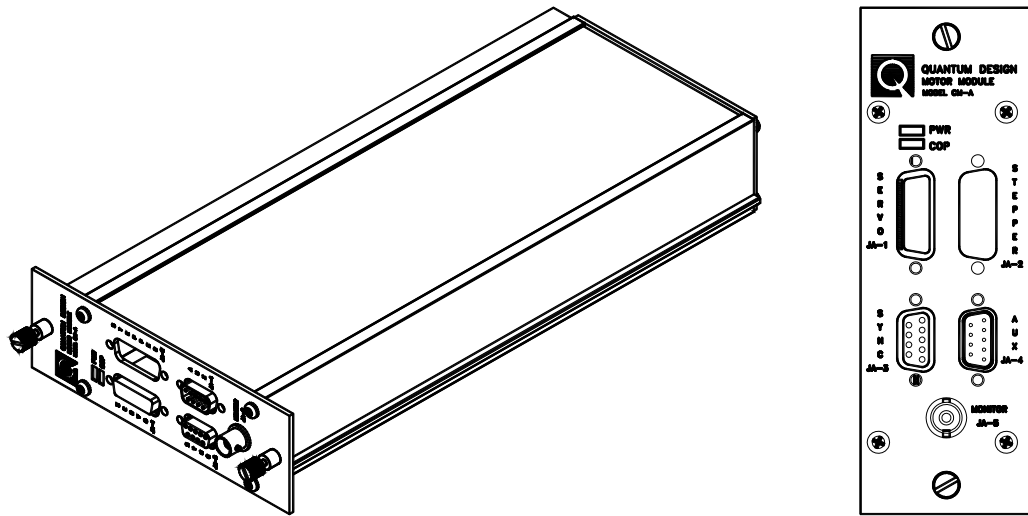


Figure A-1. Model CM-A VSM motor module (4101-100)

A.2.1 Functional Block Diagram

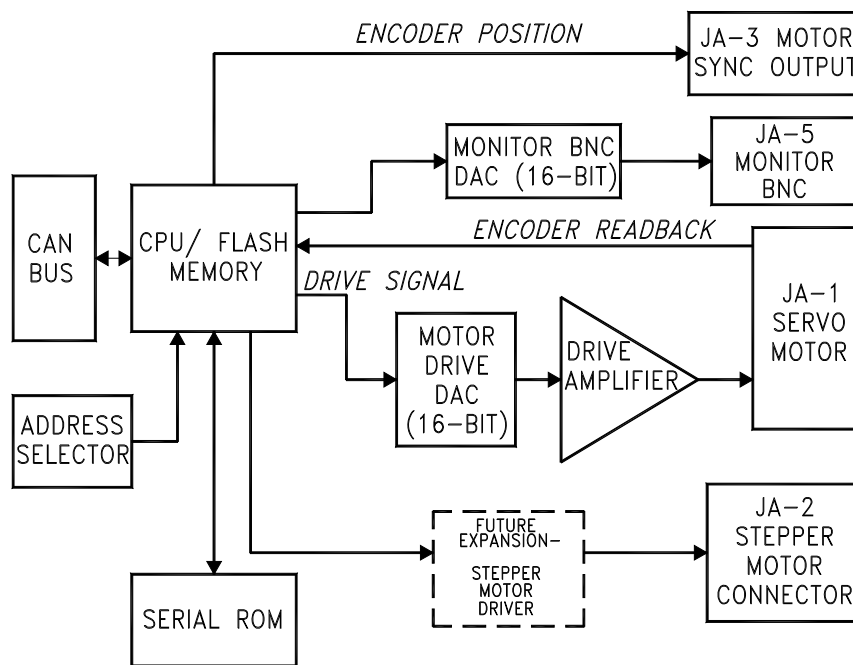


Figure A-2. Abridged functional block diagram of Model CM-A VSM motor-module specifications

A.2.2 Specifications

Table A-1. Electrical specifications for Model CM-A VSM motor module

Drive Current Limit	3 A
Drive Voltage Compliance Limit	20 V peak
Encoder Range	32-bit
Supply Voltage	±24 V DC

A.3 Model CM-A VSM Motor Module: Front Panel

A.3.1 Indicator LEDs

The front panel of the Model CM-A VSM motor module has two LEDs in the top left, as shown in Figure A-1. The PWR LED indicates the power-on status of the module. The COP (CANopen Protocol) LED indicates the status of the CAN network controller. Table A-2 outlines the LED states and provides solutions in the event of a problem.

Important: The error information in Table A-2 refers to situations that persist for longer than about 15 seconds. Typically, when the module is powered on, the LEDs may briefly flash red before they turn green. This is a normal part of the startup or reset sequence.

Table A-2. LED guide for Model CM-A VSM motor module

LED	COLOR	STATUS	MEANING AND/OR SOLUTION
PWR	Green	On	The processor is running with no errors (normal).
	Red	Flashing	Errors were encountered during the self-test. The flashing sequence can be used to determine the cause of the failure.
COP	Green	On	CAN status is operational (normal).
		Flashing	CAN status is pre-operational. Verify that cable is connected to PC.
	Red	On or flashing	Error on the CAN bus. Contact Quantum Design for assistance.

If you are unable to achieve operation with both LEDs green, please contact Quantum Design for assistance.

A.3.2 Connectors and Pinout Tables

A.3.2.1 JA-1: SERVO CONNECTOR

This connector is used to provide the current drive to the motor and read back the position information from the encoder. This connector also supports serial communication to logic associated with the motor (e.g., serial ROM for storing calibration or configuration information about the motor).

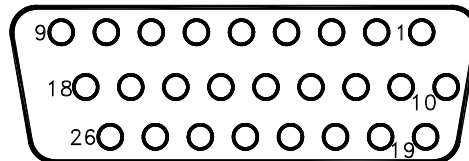


Figure A-3. JA-1: Servo connector for the Model CM-A VSM motor module

Table A-3. JA-1: Servo connector for the Model CM-A VSM motor module

PIN	FUNCTION
1	Motor -
2	+5 V
3	Encoder Input A+
4	Encoder Input B+
5	Unused Encoder Input Z+
6	n.c.
7	n.c.
8	Motor Max Limit Switch
9	Motor Min Limit Switch
10	Motor +
11	Ground
12	Encoder Input A-
13	Encoder Input B-
14	Unused Encoder Input Z-
15	n.c.
16	n.c.
17	Motor Max Limit Switch Rtn
18	Motor Min Limit Switch Rtn
19	Serial Com Data Out
20	Serial Com Data Input
21	Serial Com Clock
22	Serial Com Select 1
23	Serial Com Select 2

A.3.2.2 JA-2: STEPPER CONNECTOR

The stepper connector is available for future expansion. You must contact Quantum Design before you attempt to use the connector.

A.3.2.3 JA-3: MOTOR SYNC CONNECTOR

This connector outputs the motor encoder position as a high-speed digital serial signal. This would normally be connected to a synchronous detection module such as the Model CM-B VSM module.

Important: This sync connector and the short crossover sync cable (3096-400) to module CM-B are no longer used in newer versions of firmware in modules CM-A and CM-B (after ca. 2009).

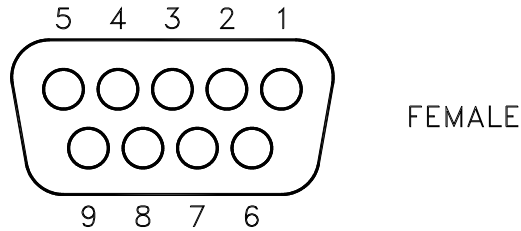


Figure A-4. JA-3: Motor sync connector for the Model CM-A VSM motor module

Table A-4. JA-3: Motor sync connector for the Model CM-A VSM motor module

PIN	FUNCTION
1	Sync +
2	Data +
3	Clock +
4	PSync +
5	Ground
6	Sync –
7	Data –
8	Clock –
9	PSync –

A.3.2.4 JA-4: AUX CONNECTOR

This connector provides three analog inputs and three digital I/O lines for future options.

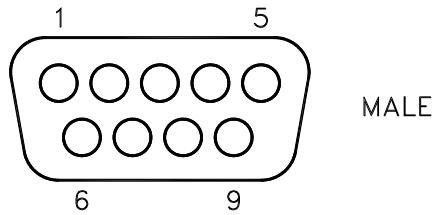


Figure A-5. JA-4: Aux connector for the Model CM-A VSM motor module

Table A-5. JA-4: Aux connector for the Model CM-A VSM motor module

PIN	FUNCTION
1	+5 V
2	Digital I/O P3.10
3	n.c.
4	Analog Input P5.13
5	Ground
6	Digital I/O P3.11
7	Digital I/O P3.8
8	Analog Input P5.12
9	Analog Input P5.14

A.3.2.5 JA-5: MONITOR BNC

This connector is for use by Quantum Design. It can be configured to output signals for diagnostic purposes. By default, the firmware is configured to output the motor position here.

A.4 Model CM-A VSM Motor Module: Rear Panel

The rear panel of the module contains an address selector, a single-guide hole, and the CAN connector through which the module sends and receives network data and receives power.

A.4.1 Address Selector

Each module on the CAN bus must have a unique 5-bit binary address. The selector on the back panel is used to set the four least significant bits, and an internal jumper sets the most significant bit. If the selector is set to zero (0), the module uses its default address. For a Model CM-A VSM motor module, the default address is 10 (or equivalently, “A” on the selector).

A.4.2 Single Guide Hole

The single guide hole is used to align the connector with one of the back-row (high-power) receptacles on the Model 1000 modular control system.

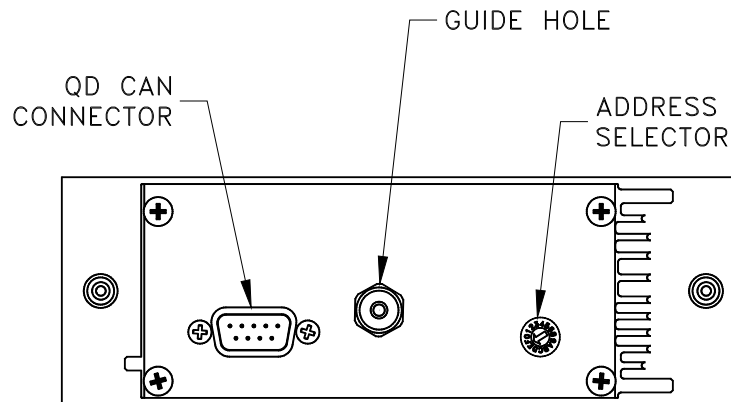


Figure A-6. Rear panel of the Model CM-A VSM motor module

A.4.3 QD CAN Connector

The QD CAN connector is the main communication connection for controlling the module. The CAN network signals (CAN High, CAN Low) are connected to all other CAN modules on the bus and to the PC. Power (± 24 volts), reset, and sync signals are also provided to the module through this connector.

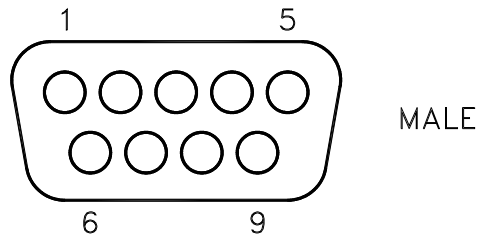


Figure A-7. Quantum Design CAN connector for the Model CM-A VSM motor module

Table A-6. QD CAN connector on the rear of the Model CM-A VSM motor module

PIN	FUNCTION
1	-24 V
2	CAN Low
3	Power Return (24V)
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High / Reset
9	+24 V DC

A.5 Maintenance

(Service Note 1096-307)

The CAN motor module CM-A (QD part number 4101-100), which is used to drive the linear motor of the VSM option, dissipates a significant amount of heat when providing the large currents (sometimes over 1 amp) to the motor drive coil. Therefore, it is critical that adequate air cooling is supplied by the fan on the back of the Model 1000 Modular Control Center. If the motor module overheats, the current output is turned off abruptly until the amplifiers cool sufficiently, at which point the current is turned back on. This causes erratic behavior of the motor and presents a hazard to the user. It might also result in damage to the hardware or your research sample.

To help prevent such overheating effects, please follow these guidelines:



WARNING!

As with any CAN module, turn off the power to the Model 1000 before inserting or removing the motor module.

- Regularly—every few months—clean the filter of the air intake fans on the back of the Model 1000. In the case of the upper fan for the module cooling (this is the most critical fan), remove the filter by first pulling off the black plastic guard. The lower fan, for the power supply drawer, can be cleaned by turning off the Model 1000 and vacuuming the outside of the filter.
- Maintain a laboratory temperature below 25 °C.
- Keep the Model 1000 at least 25cm away from walls so that air flow is not impeded.
- Cooling air inside the Model 1000 flows upward past the module and exits at the grate by the front plate of the modules. Make sure these grates are unobstructed.
- Try to minimize the dust level in the lab and keep the floors clean.
- Connect all cables before activating the VSM option, and do not unplug the motor drive cable while the option is activated. Reconnecting the cable in this state can lead to motor malfunction.

Quantum Design is continually working to improve the handling of error conditions such as this by enhancing the software, module firmware, and module hardware. Updates to option software (such as the PPMS VSM option), new service notes, and application notes are posted on our website www.qdusa.com. Firmware and hardware updates are handled on an individual basis by Quantum Design service.

If you are encountering performance problems with your motor module after observing the above maintenance steps, please contact your local Quantum Design service representative.

Model CM-B VSM Detection Module

B.1 Introduction

This appendix contains the following information:

- Section B.2 provides a functional overview of the Model CM-B VSM detection module, including a block diagram and electrical specifications.
- Section B.3 describes the front panel and relevant components of the Model CM-B VSM detection module.
- Section B.4 describes the back panel and relevant components of the Model CM-B VSM detection module.

B.2 Functional Overview

The Model CM-B (4101-150) is a synchronous detection module that performs the real-time signal processing for the VSM option. The module and its front panel are shown in Figure B-1.

The principle function of this module is to detect the in-phase and quadrature-phase components of one or two input signals (e.g., pickup coils), as well as a digital reference from, say, a position encoder. The detection is done by multiplying each of the signals by both a sine function and a cosine function. These sine components are computed once per cycle and can be output at this rate, or they can be averaged for multiple cycles with statistics calculated for the ensemble of measurements. Other features include a thermometer bridge circuit for temperature measurements, two programmable gain amplifiers, in-system programmable on-board flash memory for program storage, and a serial ROM for calibration and other configuration data.

The module is designed to plug into the Model 1000 modular control system or an equivalent host chassis that can provide power and the required CAN network signals to communicate with the module.

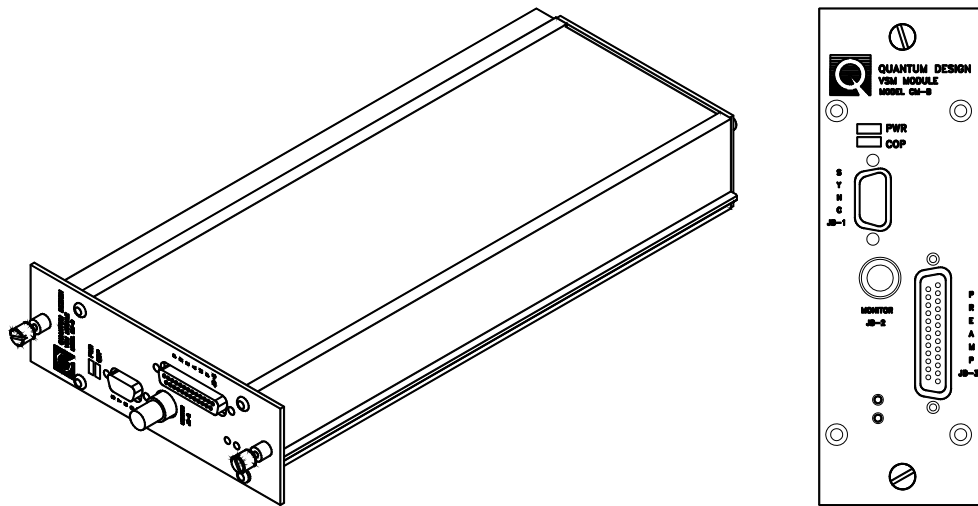


Figure B-1. Model CM-B VSM detection module (4101-150)

B.2.1 Functional Block Diagram

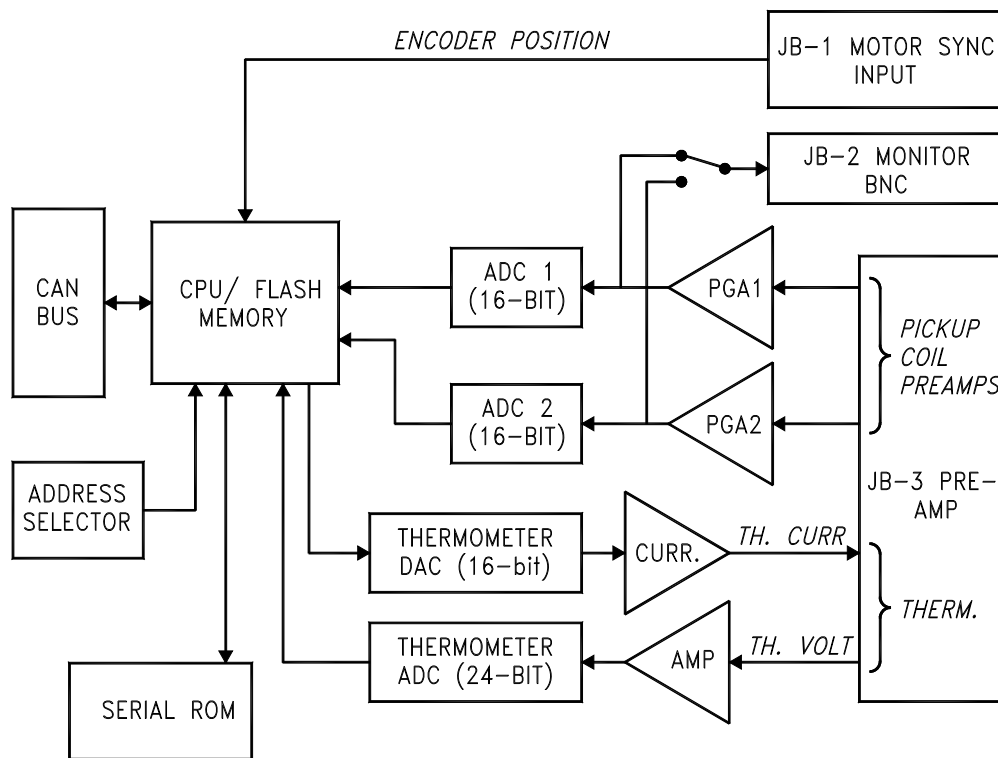


Figure B-2. Abridged functional block diagram of the Model CM-B VSM detection module

B.2.2 Specifications

Table B-1. Electrical specifications for the Model CM-B VSM detection module

Input Ranges (from preamp)	5 V, 0.5 V, 50 mV, 5 mV
Thermometer Current Ranges	±0.02 mA, ±0.5 mA
Thermometer Voltage Range	±10 mV
Thermometer Sample Rate	14 Hz
Thermometer Resistance Range	20 to 200,000 ohms
Supply Voltage	±24 V DC

B.3 Model CM-B Detection Module: Front Panel

B.3.1 Indicator LEDs

The front panel of the Model CM-B detection module has two LEDs in the top left, as shown in Figure B-1. The PWR LED indicates the power-on status of the module. The COP (CANopen Protocol) LED indicates the status of the CAN network controller. Table B-2 outlines the LED states and provides solutions in the event of a problem.

Important: The error information in Table B-2 refers to situations that persist for longer than about 15 seconds. Typically, when the module is powered on, the LEDs briefly flash red before they turn green. This is a normal part of the startup or reset sequence.

Table B-2. LED guide for the Model CM-B VSM detection module

LED	COLOR	STATUS	MEANING AND/OR SOLUTION
PWR	Green	On	The processor is running with no errors (normal)
	Red	Flashing	Errors were encountered during the self-test. The flashing sequence can be used to determine the cause of the failure.
COP	Green	On	CAN status is operational (normal)
		Flashing	CAN status is pre-operational. Verify that cable is connected to PC.
	Red	On or flashing	Error on the CAN bus. Contact Quantum Design for assistance.

If you are unable to achieve operation with both LEDs green, please contact Quantum Design for assistance.

B.3.2 Connectors and Pinout Tables

B.3.2.1 JB-1: MOTOR SYNC CONNECTOR

This connector reads the motor encoder position from the Model CM-A VSM motor module as a high-speed digital serial signal.

Important: This sync connector and the short crossover sync cable (3096-400) to module CM-A are no longer used in newer versions of firmware in modules CM-A and CM-B (after ca. 2009).

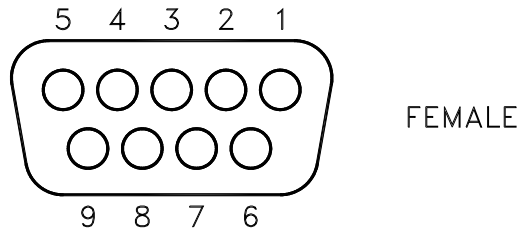


Figure B-3. JB-1: Motor sync connector for the Model CM-B VSM detection module

Table B-3. JB-1: Motor sync connections for the Model CM-B VSM detection module

PIN	FUNCTION
1	Sync +
2	Data +
3	Clock +
4	PSync +
5	Ground
6	Sync -
7	Data -
8	Clock -
9	PSync -

B.3.2.2 JB-2: MONITOR BNC

This connector outputs the amplified pickup coil signal.

B.3.2.3 JB-3: PREAMP CONNECTOR

This connector is the main connection to the preamplifiers and coilset puck. It contains two analog inputs for the synchronous detection, current and voltage for a thermometer, power for the preamp, and serial communications to the serial ROM in the preamp box for storing calibration or configuration information.

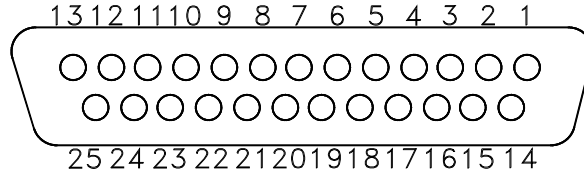


Figure B-4. JB-3: Preamp connector for the Model CM-B VSM detection module

Table B-4. JB-3: Preamp connector for the Model CM-B VSM detection module

PIN	FUNCTION
3	Serial Com Select
4	Serial Com Data MOSI
5	Thermometer Current +
6	Thermometer Voltage +
9	+15 V
10	Channel 1 Input +
11	+5 V
12	Channel 2 Input +
13	Signal Ground
16	Serial Com Clock
17	Serial Com Data MISO
18	Thermometer Current -
19	Thermometer Voltage -
22	-15 V
23	Channel 1 Input -
25	Channel 2 Input -

B.4 Model CM-B Detection Module: Rear Panel

The rear panel of the Model CM-B VSM detection module contains an address selector, two guide holes, and the CAN connector through which the module sends and receives network data and receives power.

B.4.1 Address Selector

Each module on the CAN bus must have a unique 5-bit binary address. The selector on the back panel is used to set the four least significant bits, while an internal jumper sets the most significant bit. If the selector is set to “0,” the module uses its default address. For a Model CM-B VSM detection module, the default address is 8.

B.4.2 Guide Holes

The two guide holes are used to align the connector with either a low-power receptacle or a high-power receptacle on the Model 1000 modular control system.

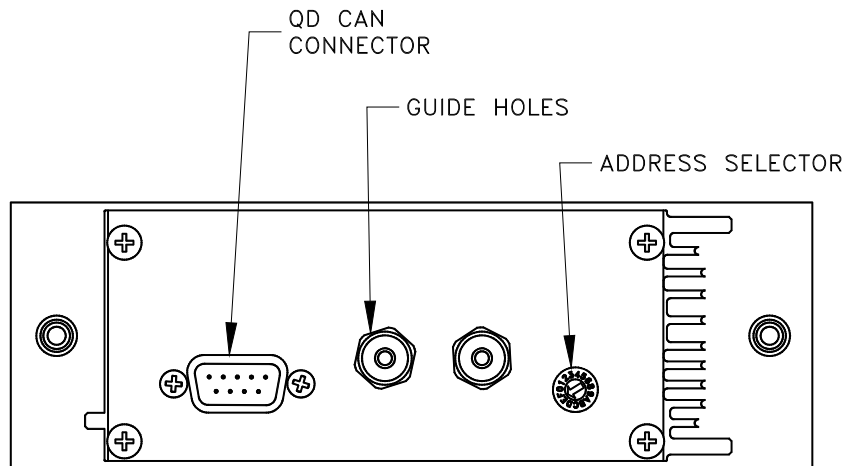


Figure B-5. Rear panel of the Model CM-B VSM detection module

B.4.3 QD CAN Connector

The QD CAN connector is the main communication connection for controlling the Model CM-B VSM detection module. The CAN network signals (CAN High, CAN Low) are connected to all other CAN modules on the bus and to the PC. Power (± 24 volts), reset, and sync signals also are sent to the module through this connector.

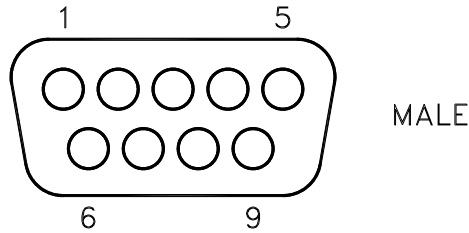


Figure B-6. QD CAN connector on rear of the Model CM-B VSM detection module

Table B-5. QD CAN connector on the rear of the Model CM-B VSM detection module

PIN	FUNCTION
1	-24 V
2	CAN Low
3	Power Return (24V)
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High / Reset
9	+24 V DC

Application and Service Notes: VSM

Application notes expand upon the technical content of the user's manual. They serve the function of training the user on topics such as sample mounting, measurement protocol and data interpretation, and also of warning the user about understanding and preventing artifacts in the resulting data. Service notes, by contrast, outline procedures for instrument maintenance, testing, and modification. Note that existing application and service notes have been incorporated into this version of the manual and those include:

Application Notes:

- [VSM Sample Mounting Techniques](#) (11/10) - 1096-306
- [Sample Centering and Temperature-Dependent Magnetization Measurements using the PPMS® VSM](#) (6/10) - 1096-305
- [Mounting Samples Loosely Causes Moment Noise in VSM Measurements](#) (9/04) - 1096-303
- [Performing VSM Measurements in PPMS® High-Field \(14-T or Higher\) Magnets](#) (11/03) - 1096-301

Service Notes:

- [Operating Guidelines for the Motor Module used for VSM](#) (6/10) - 1096-307

This space is reserved for any relevant application and service notes that have been added after the release of this manual. Please check the Quantum Design website www.qdusa.com regularly for technical note updates and new postings.

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



VSM Oven Option User's Manual

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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
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
Canada 2,089,181 High Symmetry DC Squid System
Japan 2,533,428 High Symmetry DC Squid System
Japan 2,533,428 High Symmetry DC Squid System

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P R E F A C E

Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 provides an overview of the scope of the manual.
- Section P.3 outlines the contents of the manual.
- Section P.4 shows the conventions that appear in the manual.

P.2 Scope of the Manual

This manual contains background about the Vibrating Sample Magnetometer (VSM) Oven option; instructions for installing and using the VSM Oven software and hardware; and instructions for performing sensitive measurements when the VSM Oven option is installed.

P.3 Contents of the Manual

- Chapter 1 provides an overview of the VSM Oven option, the theory of operation, safety recommendations, and contact information for Quantum Design..
- Chapter 2 provides instructions for installation and removal of the VSM Oven option.
- Chapter 3 covers sample composition and how to mount samples for measurement with the VSM Oven.
- Chapter 4 summarizes the measurement process with the VSM Oven option and guides you through an immediate-mode measurement.
- Chapter 5 describes the hardware and electrical components of the VSM Oven option and the contents of the VSM Oven Option User's Kit.

- Chapter 6 describes the differences you will encounter when using MultiVu with the VSM Oven option compared to the standard VSM equipment.
- Appendix A provides a functional description of the Model CM-C VSM Oven module, including diagrams and electrical specifications.
- Index is a guide to information organized by key terms and phrases.

P.4 Conventions in the Manual

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

CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.



WARNING!

Text is set off in this manner to signal conditions that could result in bodily harm or loss of life.



WARNING!

Text is set off in this manner to signal electrical hazards that could result in bodily harm or loss of life.

Introduction to the VSM Oven Option

1.1 Introduction

This chapter contains the following information:

- Section 1.2 is an overview of the VSM Oven option.
- Section 1.3 describes notable features of the VSM Oven option.
- Section 1.4 describes the VSM Oven option theory of operation.
- Section 1.5 outlines safety considerations for working with the VSM Oven option.
- Section 1.6 lists contact information at Quantum Design and its service centers.

1.2 Overview

The Oven for the VSM provides a way to make sensitive D.C. magnetometry measurements at controlled temperatures from 300 K up to 1000 K. Using a specially designed heated sample holder and VSM sample rod with electrical feedthrough, the VSM Oven vibrates the sample inside the VSM detection coilset. Both the standard VSM option and a high-vacuum option are required in order to operate the VSM Oven.

Heating of the sample is achieved by applying current to a Platinum resistive heating element lithographically patterned onto the custom-designed sample holder. A thermocouple embedded on the back side of the sample holder measures the temperature in the sample region, and a thermistor at the top connector of the sample holder corrects for heating of the cold junction. In this manual, we will refer to the VSM Oven sample holder as the *heater stick* (see Figure 5-1). To maximize thermal contact, the sample is attached to the heater stick using alumina-based cement (included in VSM Oven Option User's Kit, Figure 5-5). Then, the platinum heater region is wrapped securely with a thin copper-foil radiation shield that retains heat and reduces thermal gradients between the sample and heater stick.

The VSM Oven heater region is a 1 inch long heated substrate on which the sample is glued. This design is in contrast to that of other high-temperature oven inserts for magnetometers, which often are bulky and have long thermalization times. By embedding the thermometry in the substrate along with the heater, we have made vast improvements on typical thermal-response times and sample-temperature accuracy. Due to the low thermal mass of the heated substrate, you can maintain heating rates at the sample of over +200 K/min throughout the full temperature range of the oven. Note that the cooling rate will depend on the current temperature of the heater stick, because cooling occurs through thermal radiation from the heater stick to the VSM detection coilset.

Ease of use is another advantage of the Quantum Design VSM Oven option—it uses the same VSM detection coilset as the standard (low temperature) mode of the VSM option. To change from the standard VSM mode to the VSM Oven mode, you simply insert a different sample rod into the VSM.

1.3 Notable Features of the VSM Oven Option

Figure 1-1 illustrates the hardware and functional connections necessary for operating the VSM Oven. A new CAN module, the Model CM-C VSM Oven module, has been introduced to handle the temperature control of the VSM Oven heater stick. This module plugs into the Model 1000 Modular Control System, which communicates with the PC via the CAN network cable. The oven-control cable plugs into the front panel of the oven-control module. This cable passes heater power and temperature readback information to the heater stick via the electrical feedthrough on the wired access port and the wired oven sample rod.

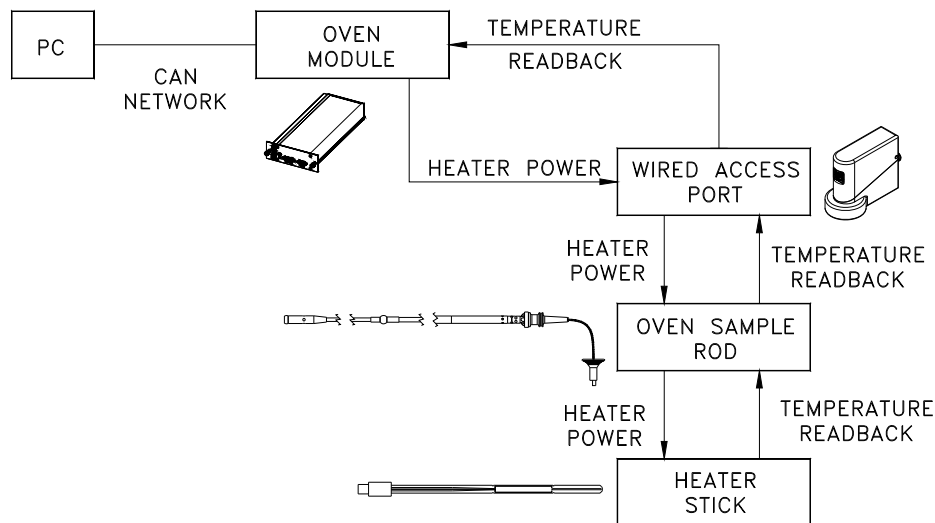


Figure 1-1. Operating principle for the VSM Oven option

1.4 Theory of Operation

The platinum heater is driven synchronously to the power line frequency (50 Hz or 60 Hz, depending on country and region) with a sinusoidal excitation. Magnetic feedthrough from the heater currents into the VSM detection coilset is minimized by patterning the heater noninductively and by operating at line frequency, where the VSM detection circuitry can easily reject the signal. The thermocouple voltage is monitored constantly by a sensitive low-drift DC microvolt preamp in the VSM Oven module. The thermocouple junction is located in the middle of the heater region of the stick, and the thermocouple wires terminate at the connector at the top of the stick. Note that the thermocouple table is referenced to a cold junction at 273 K (0 °C). Also located at the connector is a negative temperature coefficient thermistor that corrects for the

thermocouple cold junction temperature. The thermistor is read about 10 times a second using a 2 msec current pulse.

The temperature at the sample T_{sample} is calculated from the thermocouple voltage ΔV_{TC} and thermistor resistance $R_{thermistor}$ as follows:

$$T_{sample} = T_{TC}(\Delta V_{TC} + \Delta V(T(R_{thermistor}))),$$

where

$T(R_{thermistor})$ = standard table for temperature vs. thermistor resistance,

$\Delta V(T)$ = standard table for type S thermocouple voltage vs. temperature standard, and

$T(\Delta V)$ = inversion of the $\Delta V(T)$ thermocouple table.

In order to enable temperature control down to 300 K at the heater stick, the VSM puck is set to 295 K while the system operates in VSM Oven mode. Due to the heat load from the heater stick when it is at high temperatures, the software lets the block temperature go as low as 283 K while it tries to maintain the puck temperature at 295 K.

Figure 1-2 below shows a simplified schematic of the control circuit for the heater stick. "PID" refers to the Proportional-Integrator-Differential temperature control of the A.C. drive for the heater.

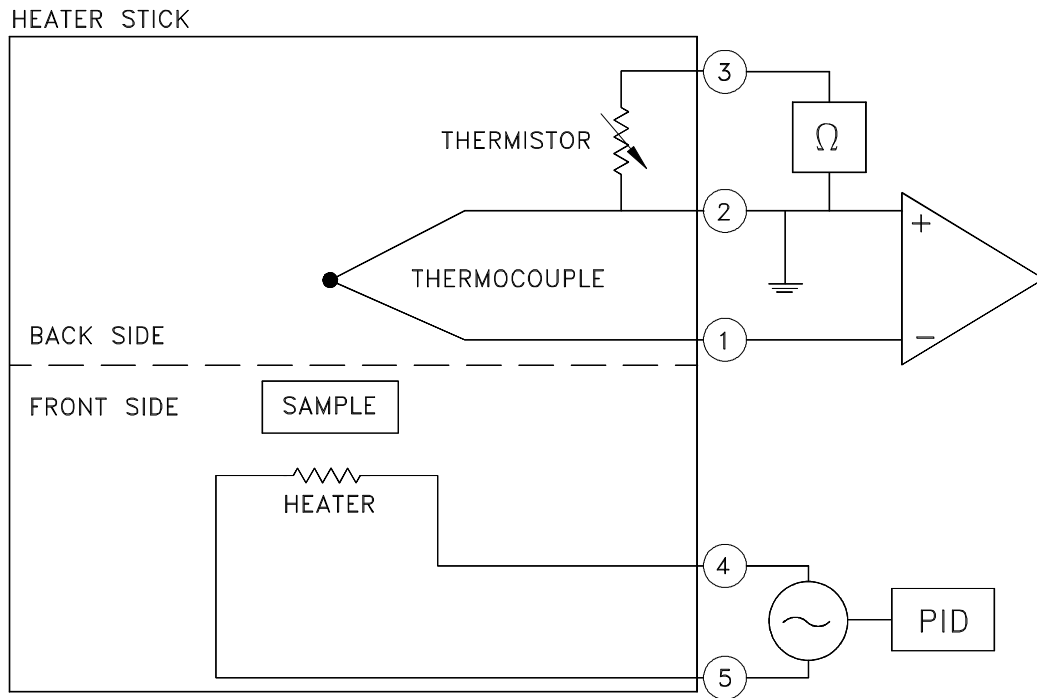



Figure 1-2. Control circuit for the VSM Oven heater stick

1.5 Overheating Prevention Mechanisms

To safeguard the equipment from overheating, the coilset temperature is set to 295 K while it is operating in oven mode, and the VSM Oven software uses the VSM coilset temperature as the primary diagnostic. In the event that the VSM coilset temperature rises above 350 K, the system shuts down the power to the oven heater.

1.6 Safety Precautions

	<p>WARNING!</p> <p>The VSM Oven option is used in conjunction with the Physical Property Measurement System (PPMS) family of systems, so you should be aware of the safety considerations for that equipment. PPMS-related safety precautions may include those for the use of superconducting magnets and the use of cryogenic materials (if applicable).</p>
---	---

Above all, Quantum Design and its staff ask that you use standard safe laboratory procedures.

- ❖ Use common sense.
- ❖ Pay attention to the state of the system and to your surroundings.
- ❖ Investigate and take appropriate action if the behavior of the system appears abnormal—something could be wrong with it.
- ❖ Supervise inexperienced users and train them in laboratory safety and in general electrical safety procedures.


The VSM has safety features to prevent accidents from causing injury or serious equipment damage. *If you use the equipment in a manner that is not specified by Quantum Design, the protection afforded by the equipment may be impaired.*

1.6.1 VSM High-Temperature Heater

Handle the heater stick with caution when you remove it from the cryostat.

The heater stick should be near room temperature by the time you take it from the wired access port—it has very low mass (less than 1 gram of hot material) and cools substantially when the sample chamber is vented with helium gas. Yet, you should remember that the central region of the heater stick can reach temperatures up to 727 °C (1000 K), and this would cause severe burns if you touched it.

1.6.2 Cryogenics




WARNING!

Always wear protective clothing and ensure that the room has good ventilation when you work with cryogenic materials such as liquid helium and liquid nitrogen. These precautions will help protect you against cryogenic material hazards: (1) they can expand explosively when exposed to room temperature; (2) they can cause serious burns.

- ❖ Always wear protective clothing, including thermal gloves, eye protection, and covered shoes, when you work with liquid helium, liquid nitrogen, or other cryogenics. Avoid loose clothing or loose fitting gloves that could collect cryogenic liquids next to the skin. The extreme cold of liquid and gaseous cryogenics can cause serious burns and has the potential to cause loss of limbs.
- ❖ Work with cryogenic materials only in well-ventilated areas. In the event a helium container ruptures or there is a helium spill, vent the room immediately and evacuate all personnel. In a poorly ventilated area, helium can displace the air, leading to asphyxiation. Because helium rises, it is generally safest to work with it in well-vented rooms with high ceilings.

1.6.3 Magnets



WARNING!

Any person wearing a pacemaker, electrical medical device, or metallic implant must stay at least 5 m (16.5 ft.)¹ from the dewar. In addition, personnel must keep all ferromagnetic objects at least 5 m (16.5 ft.) from the dewar. Verify that magnetic fields are at zero (0) before handling the VSM linear motor transport in any way.

The following precautions should be followed to ensure the safety of personnel who work with or around a superconducting magnet. This material is covered in more depth in main system user manual.

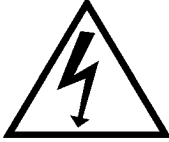
- ❖ Verify that any person who has a metallic implant or is wearing a pacemaker or electrical or mechanical medical device stays at least 5 m (16.5 ft.) from the dewar. Large magnetic fields are dangerous to anyone who has a metallic implant or is wearing a pacemaker or other electrical or mechanical medical device.

Important: The automated control system can turn on the magnet while the system is unattended. Furthermore, the three-dimensional magnetic field will penetrate nearby walls, the ceiling, and the floor. Therefore, your safety considerations should include such adjacent spaces.

¹ At the current time, 5 m should be a large enough distance to protect wearers of metallic implants or medical devices from most magnetic fields produced by Quantum Design magnets. However, the safe distance from newer magnets (in development) could be greater. Hence, personnel who work with and around the superconducting magnets should thoroughly review documentation for new equipment.

- ❖ Keep all iron, nickel, and other ferromagnetic objects at least 5 m (16.5 ft.) from the dewar. Large magnets, such as superconducting magnets, can attract iron and other ferromagnetic materials with great force.
- ❖ Never attempt to install, remove, or handle the VSM linear motor transport when there is a field set in the cryostat or in any other nearby equipment. In addition, the VSM linear motor transport must be secured when it is stored within 5 m (16.5 ft.) of the cryostat or any other large field source. The VSM linear motor transport presents a considerable hazard in a large magnetic field such as that produced by the cryostat or other laboratory equipment such as an NMR magnet, as the linear motor transport contains nearly 9 kg of iron.

1.6.4 Electricity



WARNING!

The VSM is powered by nominal voltages between 100 V to 240 V AC. These voltages are potentially lethal, so you should exercise appropriate care before opening any of the electronics units, including turning off the equipment and disconnecting it from its power source.

- ❖ Turn off and unplug all electronic equipment before removing any equipment covers.
- ❖ Keep electrical cords in good working condition and replace frayed and damaged cords.
- ❖ Keep liquids away from the workstations.

1.7 Contacting Quantum Design

If you have questions or problems related to your QD equipment, please contact your local QD service representative. See www.qd-international.com for information about your location representative. You will be asked to describe the problem, the circumstances involved, and the recent history of your system.

Installing and Removing the VSM Oven Option

2.1 Introduction

This chapter contains the following information:

- Section 2.2 includes definitions and a list of the VSM Oven components.
- Section 2.3 describes the procedures for the *initial* installation of the VSM Oven option.
- Section 2.4 describes how to install VSM Oven hardware.

2.2 Overview of the VSM Oven Installation

This chapter describes the procedures you will use to install the hardware and software for the VSM Oven option. If you purchased the VSM Oven option as part of a complete Quantum Design (QD) system, many of these procedures will have been performed before you receive the equipment.

2.2.1 Terminology

To distinguish among the various activities that are involved in installing and operating the VSM Oven option, we offer the following usages and definitions:

- *Activate option* refers to the **Utilities >> Activate Option...** command in MultiVu, which incorporates option-specific commands into MultiVu. For example, when you activate the VSM option, the **VSM Control Center** will open so that you can set VSM-related parameters.
- *Install hardware* refers to activities involved in setting up equipment, such as plugging in the VSM Oven module, connecting cables, attaching the wired access port to the VSM head, and so on.
- *Install sample* refers to inserting a sample and sample rod into the PPMS using the **VSM Install/Remove Sample Wizard**. This wizard is available only while the VSM option is activated within MultiVu.

- **“Mode”** refers to the types of VSM measurements that can be made. When you have activated the VSM option in MultiVu, you can operate in either the *standard* (low temperature) mode or the *oven* mode. During installation, the VSM Oven software is automatically incorporated into the standard VSM software, so it does not have to be activated and deactivated separately from the standard VSM option.
- **VSM Oven-enabled** refers to the VSM MultiVu application when the VSM Oven hardware and software have been integrated.
- **VSM User's Manual** refers to the *Vibrating Sample Magnetometer (VSM) Option User's Manual*.

2.2.2 VSM Oven Components

Table 2-1 lists the components of the QD VSM Oven option. Please verify that you have received them all before you start the installation.

Table 2-1. System components of the VSM Oven option

COMPONENT	PART NUMBER	ILLUSTRATION
Model CM-C VSM Oven Module*	4101-200	Figures 1-1, 5-10-5-11, A-1, A-6
Wired Access-Port Assembly	4097-020	Figures 1-1, 5-3-5-4
VSM Oven Sample-Rod Assembly	4097-010 or 4097-075	Figures 1-1, 5-2
VSM Oven-Control Cable Assembly	3097-010-01 or 3097-010-02	Figure 5-9
Heater Stick Assembly**	4097-050	Figures 1-1, 3-1, 5-1, 5-5
VSM Oven Option User's Kit	4097-040	Figure 5-5
VSM Oven Option User's Manual	1097-100	
Copper-Foil Shields**	4097-042	Figures 3-1, 5-5
VSM Oven Sample Mounting Platform Assembly**	4097-041	Figures 3-1, 5-5-5-6
Mounted Nickel Standard**	4097-055	Figure 5-5
Model 6000 Firmware ROM*		
VSM/MultiVu Application Software*		

*These items might be pre-installed. **These items are shipped in the VSM Oven Option User's Kit.

2.3 Initial Installation of the VSM Oven Option

Important: The instructions in this section are only to be used the first time the VSM Oven hardware and software is installed on the system (for example, if the oven hardware was just purchased as an upgrade to an existing system). For routine installation and removal of the oven hardware, see Section 2.4.

The VSM Oven hardware requires the following items to be present and operational on the system:

- o Standard VSM hardware and control modules, see the *VSM Option User's Manual*
- o High Vacuum option, see the *PPMS Cryopump User's Manual* (PPMS only)
- o CAN network adapter and Model 1000 Modular Control System, see the *Model 1000 User's Manual* (PPMS only)
- o Up-to-date EPROMs in the Model 6000 (version 1.914 or later) (PPMS only)

To initially install the VSM Oven option on the system, follow these steps:

- 1) Deactivate any active options and close the MultiVu program
- 2) Turn off power to the CAN control tower
- 3) Insert the VSM Oven Module CM-C into a slot in the CAN control tower
- 4) Turn on the CAN control tower
- 5) In the file C:\Qdxxx\Vsm\System\VSM.INI (where xxx is Ppms, VersaLab, or DynaCool), open in Notepad and ensure that this line exists WITHOUT any semicolon (;) at the beginning of the line:
OVEN TYPE=1 ;
- 6) Save the VSM.INI file and close Notepad
- 7) To install remaining elements of the VSM oven hardware, continue to Section 2.4.

2.4 Installing the VSM Oven Hardware

It is assumed that the VSM hardware is installed on the cryostat before the following steps are taken, see the *VSM Option User's Manual* for information on installing the VSM hardware.

As the specific hardware for the VSM Oven option is minimal, it will be simple to change from standard to oven mode. Use the Install/Remove Sample wizard in the VSM Control Center to:

- o vent the sample chamber
- o remove any existing sample rod
- o choose Oven or Standard operating mode
- o Connect the Oven Control cable (Figure 5-9) between the VSM Oven Module CM-C and the back of the wired access port
- o install the wired access port (Figure 5-4) on top of the VSM motor

Preparing Samples for the VSM Oven

3.1 Introduction

This chapter contains the following information:

- Section 3.2 describes how to prepare a sample for high temperature measurements.
- Section 3.3 describes how to mount a sample on the heater stick so that high temperature measurements can be performed.

3.2 Sample Properties

3.2.1 Size and Shape

The sample should be no wider than 3 mm (0.120 in), as the heater stick itself is 3.6 mm (0.140 in) wide. The ideal sample geometry is a thin plate that can be glued flat to the heater stick, thus allowing optimal thermal contact to the heater.

3.2.2 Composition

Solid plates and thin films are ideal for use in the VSM Oven. The sample must tolerate being bonded to the heater stick with a high-temperature adhesive. We provide a vial of alumina-based cement (see below) in the VSM Oven Option User's Kit (Figure 5-5). The sample also must be strong enough to remain intact when it is chipped off the heater stick after it has been measured.

3.3 Sample Mounting

Mount your samples on the heater stick (Figure 5-1) by using the steps below. The procedures are shown graphically in Figure 3-1, A–D. A video of this process is also available, please contact apps@qdusa.com for access to this video.

CAUTION!

Handle the heater stick by the front side, which has the grey metal film patterns. Avoid touching the back side of the heater stick where the small thermocouple wires are embedded.

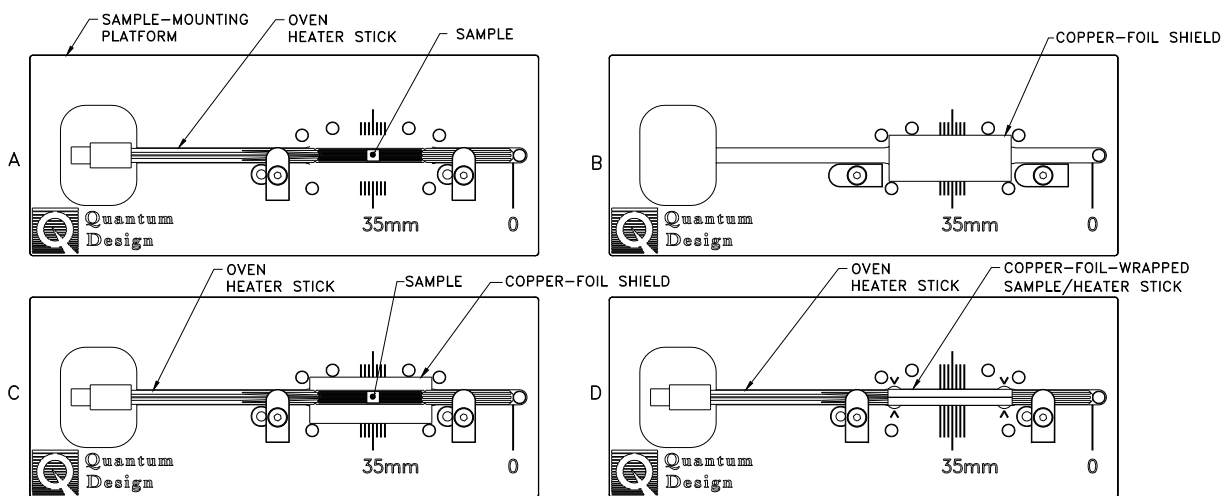


Figure 3-1. Using the sample mounting platform to mount a sample on the oven heater stick

1. Prepare the sample and heater stick and mount the sample (Figure 3-1A).
 - a. Clean the sample and the surface of the heater stick with a soft cotton swab wetted with alcohol. Clean only the patterned heater side of the stick. See Section 5.2.1 for a more detailed description of the heater stick.
 - b. After the surfaces are cleaned and dried, place the heater stick in the blue sample mounting platform supplied in the VSM Oven User's Kit (Figure 5-5). Push the bottom end of the heater stick against the plastic post located at the "0" marker, and lock it in place by rotating the white plastic tabs.
 - c. Mix the alumina cement thoroughly and apply a generous drop to the heater stick at the center of the heater meander pattern (at "35 mm" on the scale) where the sample will be placed. The cement should be thick but still fluid.
 - d. Place the sample on the glue drop before it begins to dry, pressing the sample down so that it is as close as possible to the surface of the heater. Leaving a visible border of glue around the sample, wipe away any excess cement from the heater stick.
 - e. Record the sample offset by using the scale on the mounting platform. The offset should be a value between 33 mm and 37 mm for the best temperature accuracy, as shown in Figure 3-1A.

- f. Carefully remove the heater stick from the mounting platform and cure the cement for 10 to 20 seconds by using a heat gun held about 20 cm (8 in) away. Heating the cement drives away the water base and greatly strengthens the bond.
 - g. Test the bond by gently pushing the sample from the side to verify that the sample does not easily come off the stick.
2. Wrap the sample and heater stick with a copper-foil shield (Figures 3-1B–3-1D).
 - a. Select a copper-foil shield that is shiny and free of tarnish and position it in the mounting station as shown in Figure 3-1B.
 - b. Place the heater stick with the sample on the shield (Figure 3-1C) and lock it in place with the tabs.

CAUTION!

Use a clean tool such as the tweezers in the VSM Oven Option User's Kit to handle the copper-foil shield. Do not handle the copper shield with your fingers, because it might tarnish.

- c. Using a clean hard tool such as the handle of a pair of tweezers (included in the VSM Oven Option User's Kit), push down gently on the heater region so that the copper shield begins to bend around the stick.
 - d. Fold both flaps of the shield over the top of the heater stick so that the heater and sample are completely covered (Figure 3-1D). Flatten the shield so that it is flush with the surface of the stick.
 - e. Use the tweezers to pinch the shield at both ends of the heater region so that it grips the notches in the heater stick. The locations of the notches are indicated by four “^” marks on the platform. This compression will prevent the shield from slipping during VSM measurements.
3. Remove the heater stick from the mounting platform.
 4. Plug the heater stick into the bottom end of the VSM oven sample rod.
 5. Verify that the connector for the heater stick is fully engaged with the sample rod.
 6. The sample is now ready to install in the system.

VSM Oven Measurements

4.1 Introduction

This chapter contains the following information:

- Section 4.2 summarizes the measurement process when using the Quantum Design VSM Oven option.
- Section 4.3 describes how to install samples for taking VSM Oven measurements.
- Section 4.4 describes how to take measurements with the VSM Oven option.

4.2 Overview of VSM Oven Measurements

The VSM Oven measurement process first involves locating the center position of the sample either visually or by synchronously oscillating the sample and detecting the voltage that has been induced in the coilset by the magnetized sample. This is the same procedure as for the standard VSM option.

In preparation for the measurement, you must use the automated **VSM Install/Remove Sample Wizard** to install the sample and set the system to take measurements in the VSM Oven mode.

The measurement process and use of the software are explained in the following sections. If you are experienced at taking measurements with the standard VSM, you will find that some of the MultiVu utilities and dialogs now have different capabilities and measurement limits.

Important: Before you begin the measurement process, verify that the VSM Oven option and the appropriate application software have been installed (Chapter 2) and the sample has been mounted on the heater stick (Chapter 3).

4.3 Installing a Sample

4.3.1 Attach Sample and Measure Sample Offset

Main points of the sample mounting and sample centering procedures are reviewed below. Chapter 3 has complete instructions on sample mounting—please review those procedures before you mount your sample.

4.3.2 Activate the VSM Option and the VSM Control Center

You will take measurements with the VSM Oven option by using the software for the standard VSM, which is activated and operated in MultiVu. See the *VSM Option User's Manual* for information on option activation.

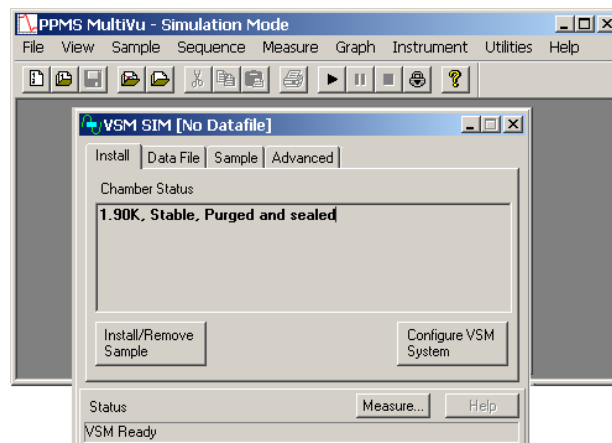


Figure 4-1. Main MultiVu window and main window of the VSM Control Center (in simulation mode)

4.3.3 Install the Sample

To install the sample you will use the **VSM Install/Remove Sample Wizard (VSM Install wizard for short)**, which is a set of automated instructions that guides you through the sample-installation process and allows you to select the VSM Oven mode or standard VSM mode.

The overall installation process involves warming and venting the sample chamber, moving the transport to the load position, choosing between operating in the standard VSM mode or the VSM Oven mode, characterizing the sample, designating a data storage file, installing the sample, and designating (or scanning for) the sample offset. This section will focus on the unique features of the VSM Install wizard in oven mode; see the *VSM Option User's Manual* for more detail about the dialogs in this wizard.

1. In the **Install** tab of the **VSM Control Center**, click on the **Install/Remove Sample** button.

2. Click on the **Open Chamber** button (below the **Instructions** area). The wizard will bring the sample chamber to room temperature, vent the chamber, and move the transport to the load position, and the **Instructions** area will show the status of these processes.
3. Now use the sequence outlined below to install the sample rod and sample. Note that you can insert or remove your sample from the cryostat at any stage of the **VSM Install** wizard, as long as the sample chamber is flooding and the transport is at the load position.
 - a. Install the wired access port on the VSM motor (Figure 5-4).
 - b. Attach the heater stick to the oven sample rod.
 - c. Open the lid on the wired access port on the VSM linear motor transport
 - d. Insert the sample rod into the wired access port until the magnetic lock at the top of the sample rod engages the magnetic lock ring in the linear motor transport. Verify that the magnetic lock has engaged the magnetic lock ring.
 - e. Plug the connector on the top of the sample rod into the plug inside the access port (see Figure 5-3).

Important: The sample will be subjected to vertical magnetic fields of up to approximately 300 gauss when it passes through the linear motor transport. If this is unacceptable for your samples, please contact Customer Service at Quantum Design.

4. Click on the **Next >>** button at the bottom of the window. This will open page 2 of the **VSM Install** wizard (Figure 4-2), in which you designate the mode of measurement (standard or oven).

Important: You can come back to this page and change your selection at any point during the **VSM Install** wizard. However, if you want to change the operating mode after the **VSM Install** wizard has ended, you must restart the wizard from the beginning.

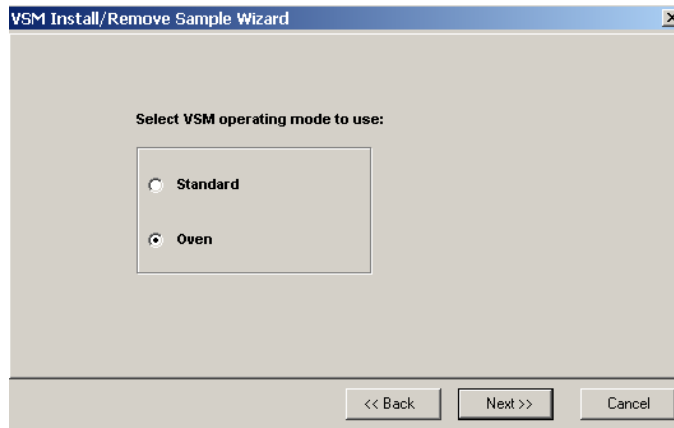


Figure 4-2. VSM Install wizard, page 2: Selecting the VSM operating mode

From this point on in the sample-installation process, you will see some differences between the instructions and dialogs for measurements in the VSM Oven mode and those for the standard VSM mode. Here we show the instructions and dialogs for the VSM Oven.

5. Click in the option button next to **VSM Oven** and then click on the **Next >>** button at the bottom of the window. Page 3 of the **VSM Install** wizard, the **Output Data File/Sample Information** dialog box, will open so you can designate an output data file.
6. Click on the **Next >>** button at the bottom of the **Output Data File** dialog box. Page 4 of the **VSM Install wizard** will open (Figure 4-3) so that you can provide or obtain the sample offset.

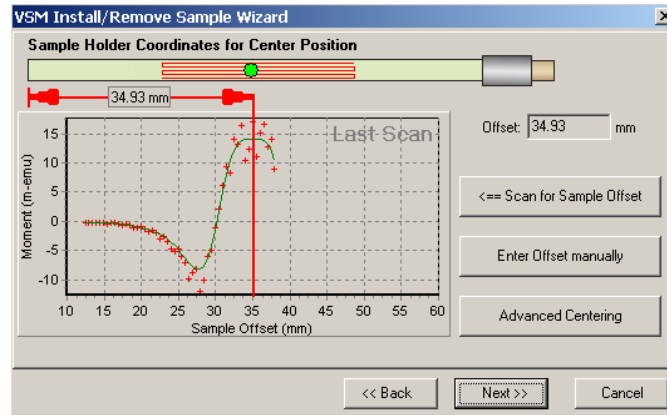


Figure 4-3. VSM Install wizard, page 4 (oven mode): Sample-holder coordinates for center position

Note that there are slight differences between the dialog boxes used for the VSM Oven mode and the standard VSM mode: for standard VSM mode, the illustration shows a sample holder, but for the VSM Oven, it shows a heater stick with a zigzag pattern that represents the heater region.

As in the case of the standard VSM sample holder, we recommend that you place the sample at a sample offset of 35 mm. The software will warn you if the sample is placed outside the heater region. The heater region includes sample offsets from 23 mm to 48 mm.

7. Click on the **Scan for Sample Offset** button if the sample signal is large enough to be detected, typically more than 10^{-5} emu. You may need to apply a magnetic field in order to create a magnetic moment. Click on **Enter Offset Manually** if you measured the offset while in the mounting platform.
8. Click on the **Next >>** button at the bottom of the dialog box. This button accepts the sample-offset value.
9. Click on the **Close Chamber** button, which is just below the **Instructions** area.
10. The **Instructions** area will indicate that a touchdown is being performed and it will report on the process as it takes place.
11. The software will then test the heater stick by measuring the resistance of the heater and thermistor and testing for continuity of the thermocouple. If all these tests succeed, this will be indicated (Figure 4-4), otherwise you must go back to the sample insertion page and check the electrical connections to the sample rod or use a different heater stick.

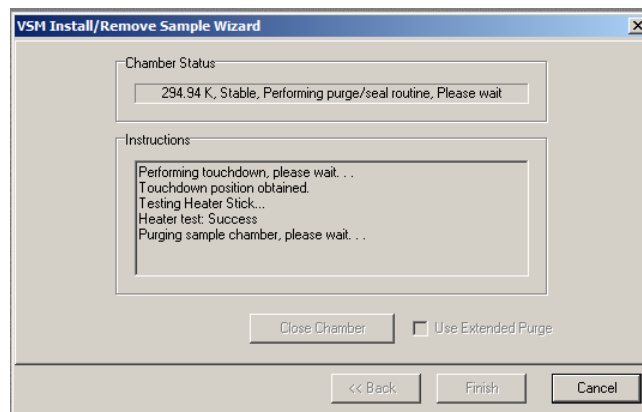


Figure 4-4. VSM install wizard, page 5: testing the heater stick.

12. The chamber will then be purged and high vacuum established. This process can take from 5 to 30 minutes, depending if the VSM hardware has recently been to high vacuum.
13. When high vacuum has been reached, the **Instructions** area will say "Sample Installation complete" and "Press 'Finish'."
14. Click on the **Finish** button at the bottom of the window. This button will return you to the **Install** tab of the **VSM Control Center**, which now will display the system temperature and other information. The **Status** area will display "VSM Oven Active" and "VSM Oven selected" (Figure 4-5).

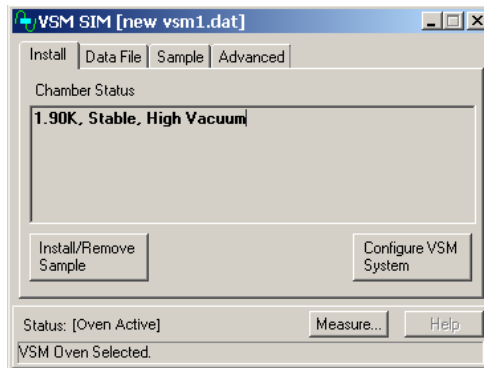


Figure 4-5. Install tab of the VSM Control Center after the VSM Oven mode has been selected.

15. You can now take measurements with the VSM Oven option.

4.4 Taking VSM Oven Measurements

Just like with the standard VSM mode, you can perform VSM Oven measurements by using immediate-mode commands (e.g., from command buttons or dropdown menus) or by constructing sequence files. See the *VSM Option User's Manual* for guidance in making measurements.

There are just a few differences when taking measurements in VSM oven mode which are noted below.

4.4.1 Touchdown Centering

Touchdowns are not required in the VSM Oven mode because the thermal expansion of the heater stick causes negligible movement of the sample relative to the detection coilset. Note, though, that if you have just changed to VSM Oven mode after operating at low temperatures in standard VSM mode, there will be a slight thermal expansion of the sample chamber. Hence, you will improve accuracy by using touchdowns for approximately 30 minutes after the sample reaches room temperature. Otherwise, we recommend selecting **No Automatic Centering** (Figure 4-6).

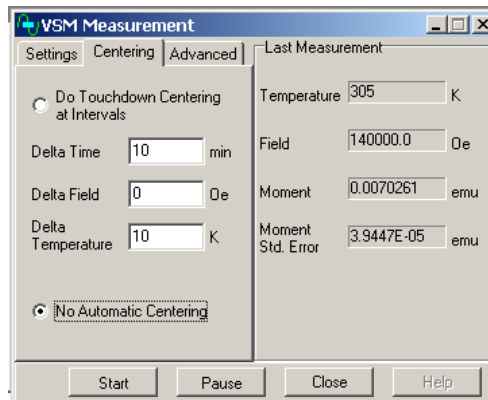


Figure 4-6. VSM Measurement dialog box: Centering tab

4.4.2 Peak Vibration Amplitude

The maximum **Peak Amplitude** is different for the standard VSM and the VSM Oven. As shown in Figure 4-7, the maximum oscillation amplitude of the VSM linear motor is 2 mm peak when the VSM Oven is operating, by comparison with 5 mm peak for the standard VSM. This is done in order to reduce heating of the linear motor drive coil in the high vacuum state.

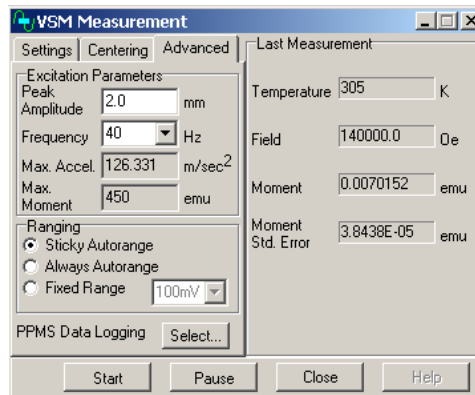


Figure 4-7. VSM Measurement dialog box: Advanced tab

VSM Oven Hardware

5.1 Introduction

This chapter contains the following information:

- Section 5.2 describes the VSM components that make up the oven option.
- Section 5.3 describes the contents of the VSM Oven User's Kit.
- Section 5.4 describes the electrical components of the VSM Oven option.

5.2 VSM Oven Hardware Components

This section describes each of the basic hardware components that make up the VSM Oven option: the oven heater stick, the oven sample rod, and the wired access port. Refer to Chapter 2 for instructions on installing the VSM Oven hardware.

5.2.1 VSM Oven Heater Stick

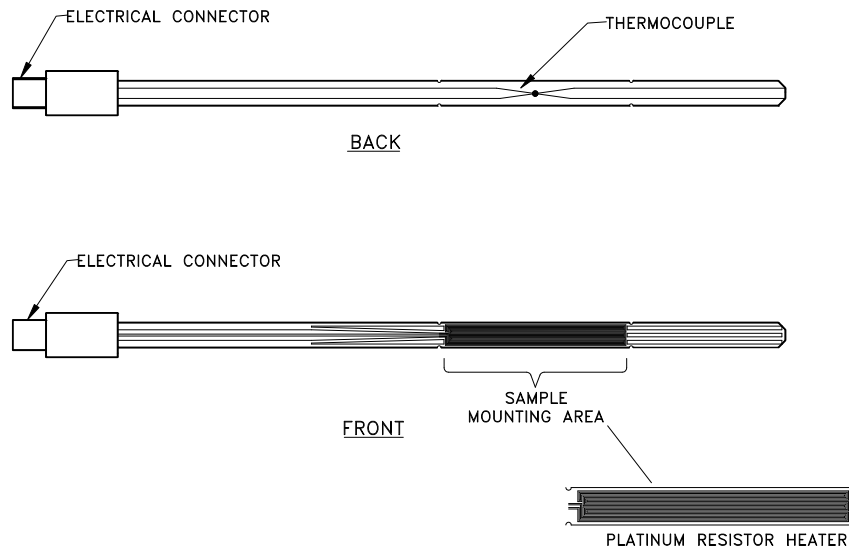


Figure 5-1. Front and back view of the VSM Oven heater stick

The VSM Oven heater stick and its parts are illustrated in Figure 5-1. It can be seen that the heater stick is a combination device that contains the heater, thermometer, and sample holder for the oven option. You will mount samples directly on the heater stick, using the alumina cement included in the VSM Oven Option User's Kit (Figure 5-5).

The heater stick is a long thin sheet of ceramic that has been silk-screened with a platinum meander pattern and then coated with a thin layer of dielectric. The ceramic has extremely low thermal conductivity, which allows the sample mounting areas to reach extreme temperatures while the electrical connections remain close to room temperature. The platinum conductor acts as the heating element of the VSM Oven option. Samples are mounted directly on top of the platinum resistor in the indicated sample mounting area.

The back of the heater stick has the temperature-detection system of the VSM Oven option. Inlaid in two grooves in the heater stick is a type S (platinum vs. platinum-10%rhodium) thermocouple. The thermocouple junction is located directly opposite the sample and platinum heater. This thermocouple provides a very precise measure of the sample temperature and it is rated for temperatures over 1000 K.

A thermistor is embedded at the top of the heater stick, inside the protective shield. The function of this thermistor is to correct the cold junction temperature of the thermocouple, because the thermocouple table is referenced to a cold junction at 0 °C.

At the top of the heater stick is the five-pin male electrical connector (Figure 5-7) that provides power for the platinum heater and has voltage leads for the thermocouple and the thermistor. The heater-stick connector plugs directly into the base of the VSM Oven sample rod shown in Figure 5-2.

5.2.2 Oven Sample Rod

The VSM Oven sample rod is specifically designed for the VSM Oven option—it is not compatible with the standard VSM option. The primary changes were introduced to accommodate and protect the necessary electrical connections to the oven heater stick.

Figure 5-2 shows the oven sample rod in the vertical position (with the magnetic lock and strain relief at the top of the rod). Beginning at the top of the figure, the oven sample rod consists of the following components:

○ Electrical Connections (Top)

The top electrical connector plugs into the feedthrough on the inside of the wired access port. It has a five-pin male connector identical to the heater connector (Figure 5-7) and an anodized aluminum rim that helps you safely grip the connector when you plug it in and remove it.

Important: Always handle the oven sample rod and its connectors by using the anodized aluminum rim of the electrical connector or the strain relief portion of the oven sample rod. Never pull on the cable while unplugging it, as you might loosen the electrical connections.

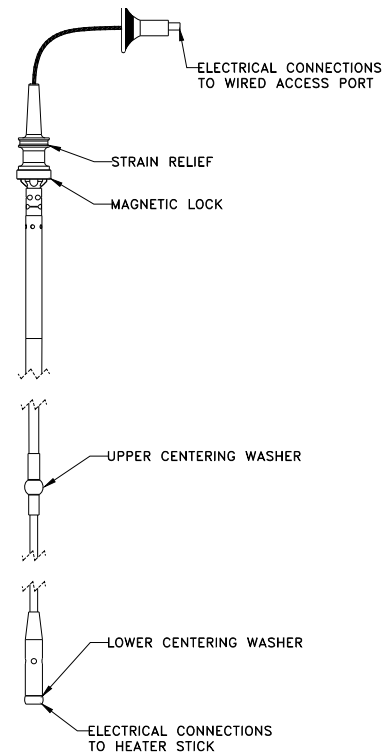


Figure 5-2. Oven sample rod

- **Strain Relief**

The strain relief portion of the oven sample rod is made of white Delrin plastic. When you install the sample rod, grip it by the strain relief portion.

This portion of the rod is designed to prevent damage to the wiring as it is fed into the shaft of the oven sample rod. It also protects the wiring while the oven sample rod undergoes vibration.

Important: Always grip the strain relief portion of the oven sample rod when you insert or remove the rod from the VSM. *Never* grip the cable of the oven sample rod while you remove it, as you might pull the electrical connections loose.

- **Magnetic Lock**

The magnetic lock is constructed of anodized aluminum. The lock contains six small, very strong magnets that attach the oven sample rod to the armature of the linear motor transport during measurement. Keep the magnets clean and prevent them from contacting any magnetic object.

- **Electrical Connections (Bottom)**

This electrical connector is the bottom of the wiring that feeds through the oven sample rod. It connects to the oven heater stick and contains a five-pin female connector.

5.2.3 Wired Access Port

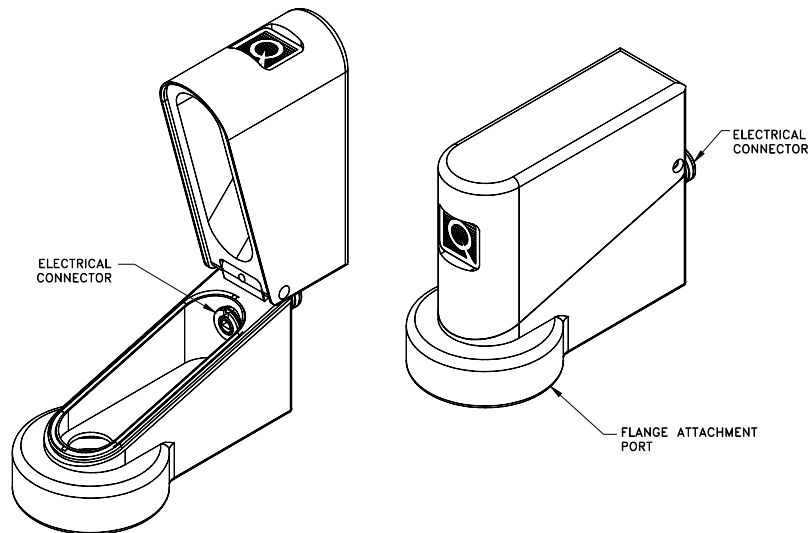


Figure 5-3. Wired access port

Figure 5-3 shows the wired access port that connects to the VSM linear motor transport. The access port is made of aluminum and is designed to be vacuum tight. The oven sample rod is connected to the oven-control cable by an electrical connector that can be seen on the back of the access port (inside and outside). This connector is specially designed: it maintains its vacuum seal and it is free to rotate. Hence, you can connect it to the oven sample rod without twisting the wiring of the rod.

On the bottom of the wired access port is the flange attachment port. This section of the access port has a hole that allows the oven sample rod to slide through and establish a magnetic lock with the VSM linear motor transport. This port screws onto the top of the VSM linear motor

transport and locking nut (Figure 5-4) and forms a vacuum-tight seal.

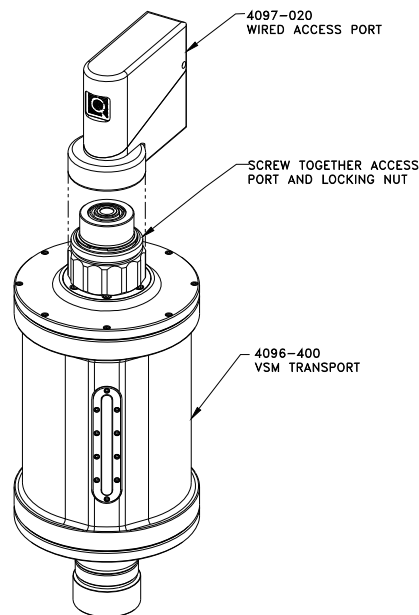


Figure 5-4. Attaching the wired access port to the VSM linear motor transport

5.3 VSM Oven Option User's Kit

The VSM Oven Option User's Kit (Figure 5-5) contains miscellaneous hardware and supplies that you will use to mount samples (see Chapter 3). The portable toolbox is a convenient way to organize these items, which are listed below.

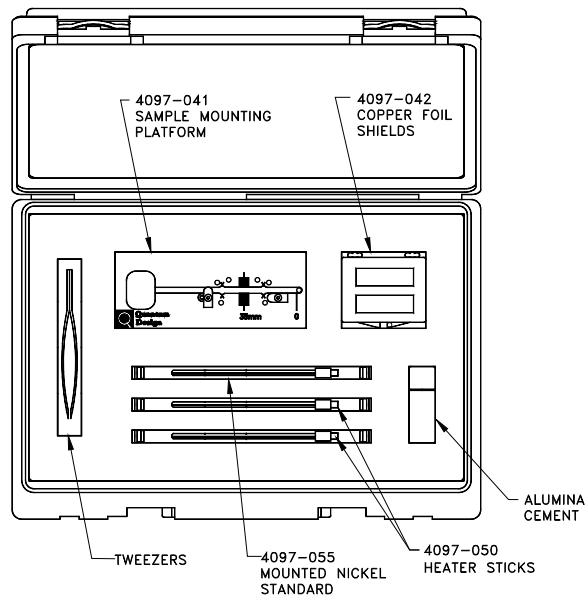


Figure 5-5. VSM Oven Option User's Kit

- **Sample Mounting Platform**

The sample mounting platform is used to mount a sample on an oven heater stick and to properly position the copper shields around the sample mounting area. Section 3.3 and Figure 5-6 provide more information about mounting samples.
- **Copper-Foil Shields**

The shields are slips of high-purity copper foil. You will wrap a shield around the sample and sample mounting area of a heater stick to provide thermal homogeneity over the sample. Refer to Section 5.3.2 for more information about how the shields are used.
- **Tweezers**

A pair of Delrin plastic tweezers is included for your use in the sample mounting process.
- **Alumina Cement**

The water-based alumina cement provides good thermal contact between the sample and heater stick. It also holds the sample firmly in place during vibration.
- **Heater Sticks**

These heater sticks are used as the heater, thermometer system, and sample holder of the VSM Oven option. The heater stick plugs into the bottom of the oven sample rod. Sections 5.2.1 and 5.4.1 and Figures 5-1 and 5-7 provide more information about the heater sticks.
- **Mounted Nickel (Ni) Standard**

The "mounted nickel standard" is an oven heater stick mounted with a small chip of pure nickel. You can verify the temperature calibration of your VSM Oven option by measuring the Curie temperature ($T_c = 627\text{ K}$) of the nickel chip.

5.3.1 Sample Mounting Platform

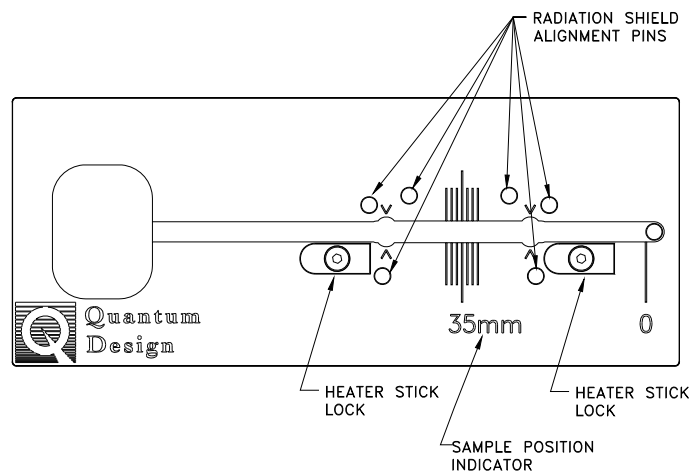


Figure 5-6. VSM Oven sample mounting platform

Shown in Figure 5-6 is the oven sample mounting platform. This platform was designed specifically for use with VSM Oven heater sticks. The heater sticks are mounted in the center groove down the long axis of the platform, with the bottom of the stick aligned at the marker labeled "0". Section 3.3 has more information about mounting a sample.

The heater stick locks are used to hold the heater stick firmly in place so that it does not slip out of position while you mount a sample. After you have positioned the heater stick properly, rotate

the locks into place. The locks also prevent the heater stick from bowing upward when you place a copper-foil radiation shield beneath it and hold the heater stick stable while you wrap the radiation shield around the stick.

The radiation-shield alignment pins are designed to fit the copper-foil radiation shields. The pins function to properly position and hold the shields in place on the heater stick while you mount a sample.

The sample-position indicator is used to determine the sample offset of the material you have just mounted on the heater stick. This is useful if the moment of your sample is very low and the VSM centering algorithm cannot locate the position of the sample. This sample-position indicator can give you a precise measurement of the sample offset that you can enter into the **VSM Install/Remove Sample Wizard**. Refer to Section 3.3 for more information about sample centering.

5.3.2 Copper-Foil Radiation Shields

The copper-foil radiation shields are thin slips of vacuum-annealed high-purity copper that has been specially selected to be free of magnetic impurities. You will wrap a radiation shield around the sample mounting area of a heater stick after you have mounted a sample.

The copper-foil radiation shields have a two-fold purpose. First, the high thermal conductivity of copper makes it an ideal medium for maintaining a thermally homogeneous region over the sample mounting area.

Important: Tightly wrap the copper-foil radiation shield around the sample and heater stick—physical contact between the copper-foil shield and the sample mounting area is important to maintaining thermal homogeneity.

The second function of the copper-foil radiation shields is to thermally isolate the heater stick and sample from the VSM coilset and the sample chamber. The copper foil has very low emissivity, which prevents excessive heat loss to the environment through radiation and allows the VSM Oven option to achieve a temperature of 1000 K.

Important: The exterior of the copper-foil radiation shields must be shiny, clean, and untarnished. Copper oxide and other materials on the outer surface of the shield can raise its emissivity, preventing the VSM Oven option from achieving temperature stability or reaching 1000 K.

5.4 VSM Oven Electronics

5.4.1 Heater-Stick Connector

Figure 5-7 shows the heater-stick connector, viewed from the outside. It attaches to the bottom of the oven sample rod, which provides a direct electrical feedthrough for each wire. The oven sample rod attaches to the wired access-port connector (Figure 5-8) with a connector identical to that in Figure 5-7. Refer to Figure 1-2 for a schematic of the heater stick.

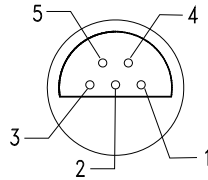


Figure 5-7. Pin assignments on the heater-stick connector

Table 5-1. Pinout connections for the heater-stick connector

PIN	FUNCTION
1	Thermocouple –
2	Thermocouple +/Thermistor B
3	Thermistor A
4	Heater A
5	Heater B

5.4.2 Wired Access-Port Connector

The wired access-port connector provides a feedthrough to the heater stick. Figure 5-8 shows the connector on the wired access port as viewed from the outside. Note that pins 4 and 5 are jumpered together inside the connector.

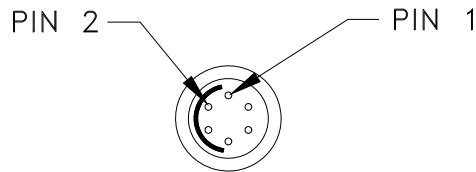


Figure 5-8. Pin assignments on the wired access-port connector (P1)

Table 5-2. Pinout connections for the wired access-port connector (P1)

PIN	FUNCTION
1	Heater A
2	Heater B
3	Thermistor A
4	Thermocouple +
5	Thermistor B
6	Thermocouple –

Table 5-3. Typical resistance values at P1 when heater stick is connected and at room temperature

PINS	RESISTANCE
1-2	200 Ω
3-5	10 k Ω
4-5	< 1 Ω
4-6	6 Ω

5.4.3 Oven-Control Cable

Figure 5-9 shows the oven-control cable (3097-010-01/-02) that connects the wired access port to the “Oven” port (JC-1) on the Model CM-C (the VSM Oven Module). The control cable provides the heater power as well as the temperature readback from both the thermocouple and the thermistor.

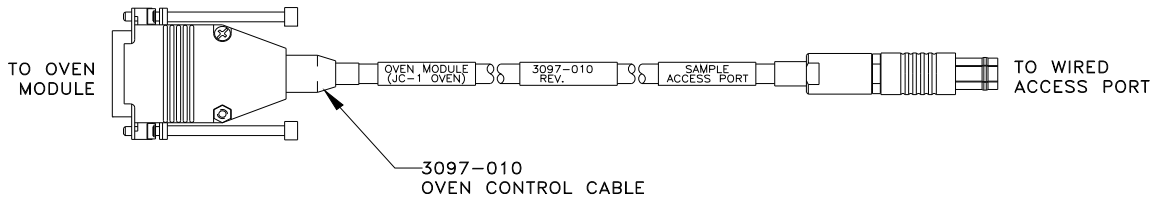


Figure 5-9. VSM Oven-Control cable

5.4.4 Model CM-C (VSM Oven Module)

Figures 5-10 and 5-11 show the front and back of the Model CM-C VSM Oven module (4101-200), which provides the heater with current and reads back temperature information from the heater stick. The oven module also handles all the temperature control for the VSM Oven option. All configuration and control of this module is through the VSM application software on the computer (PC) via the CAN-bus connector on the back panel of the module.

Appendix A contains a pinout diagram and connection table for the CAN connector.

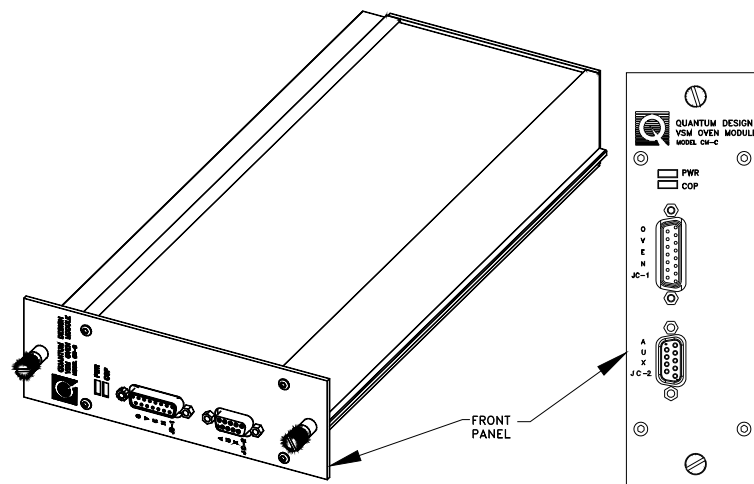


Figure 5-10. Front panel of the Model CM-C VSM Oven module

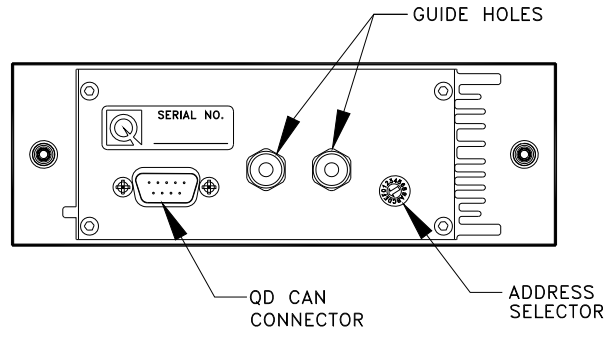


Figure 5-11. Back panel of the Model CM-C VSM Oven module

VSM Oven Software

6.1 Introduction

This document contains the following information:

- Section 6.2 summarizes the measurement process with the Quantum Design VSM Oven option.
- Section 6.3 describes some notable changes to operations associated with the VSM oven.
- Section 6.4 describes MultiVu and the VSM Oven-enabled software, focusing on changes associated with the VSM Oven.

6.2 Using the VSM Oven for Measurements

The procedures for using the Quantum Design VSM in the oven mode are almost identical to those for using it in the standard VSM mode. In fact, when you installed the VSM Oven software, it was incorporated into MultiVu as part of the standard VSM software, so you will use the same software to take measurements with the standard (low temperature) VSM and the VSM Oven. Assuming that the VSM and VSM oven hardware and software have been installed, then each time you use the **VSM Install/Remove Sample Wizard**, you will decide whether you will be measuring with the standard VSM (low temperatures) or with the VSM oven. Other than that, the same process is used for both types of measurements.

6.3 Changes Associated with the VSM Oven

6.3.1 Prevention of Overheating

To safeguard the equipment from overheating while it operates in oven mode, the cryostat sets the coilset temperature to 295 K, and the VSM Oven software uses the VSM coilset temperature as the primary diagnostic. In the event the VSM coilset temperature rises above 350 K, the system shuts down the power to the oven heater.

6.3.2 Changes to Cryopump High-Vacuum Operations

The VSM linear motor transport increases the volume of the sample chamber by comparison with options that do not use the VSM linear motor transport. Hence, when you are in VSM Oven mode, cryopump operations have been modified in the following ways:

- When a sample is installed, a purge/seal operation will be performed before the system begins any pre-pumping. This helps remove adsorbed gases and water from surfaces on the linear motor.
- After the cryopump valve has been opened, the timeout to reach high vacuum has been increased.
- In the event that the sample chamber does not reach the base pressure, the VSM oven software issues a "High Vacuum" error warning to the user and records the error in the `VsmLog.txt` file. The system does not stop VSM oven operations when there is a High Vacuum error.

6.4 MultiVu and VSM Oven-Enabled Software

This section provides an overview of MultiVu and the **VSM Control Center** when the VSM Oven has been enabled. Also discussed are changes to the standard VSM software that permit measurements with the VSM Oven option.

6.4.1 MultiVu Changes

6.4.1.1 WRITING SEQUENCES

If you have the VSM Oven hardware, MultiVu will allow you to write sequences containing temperatures up to 1000 K. In fact, you do not have to be in VSM Oven mode when you write a sequence that contains high temperatures—you can write high temperatures into a sequence even when the VSM option has not been activated. This approach streamlines the construction of high-temperature sequences, as it would be inconvenient if you could only write them while the system is in VSM Oven mode. (But remember that the VSM option must be activated before you can write sequences containing VSM-specific commands and before the system can interpret VSM sequence commands.) Then, when you activate the VSM option and run a VSM sequence, MultiVu will check the temperature limits of the sequence and abort it if the limits are not appropriate to the current VSM mode (for example, if temperatures above 400 K are requested but the system is operating in the standard, low-temperature VSM mode).

6.4.1.2 TEMPERATURE REPORT AND SETTINGS

When the VSM oven is operating, the **Temperature-System** dialog box and the **Temperature** panel of the MultiVu **Status** bar report the temperature of the oven heater stick. You can set the temperature of the VSM Oven to a value from 300 K to 1000 K by using MultiVu. The maximum recommended slew rate is 200 K/min (by comparison with 20 K/min in other options) because of

the very low thermal mass of the heater stick and the ease with which temperature can be controlled.

6.4.2 VSM Control Center Components

Figure 6-1 shows the **VSM Control Center** (in oven mode (“[oven active]”)).

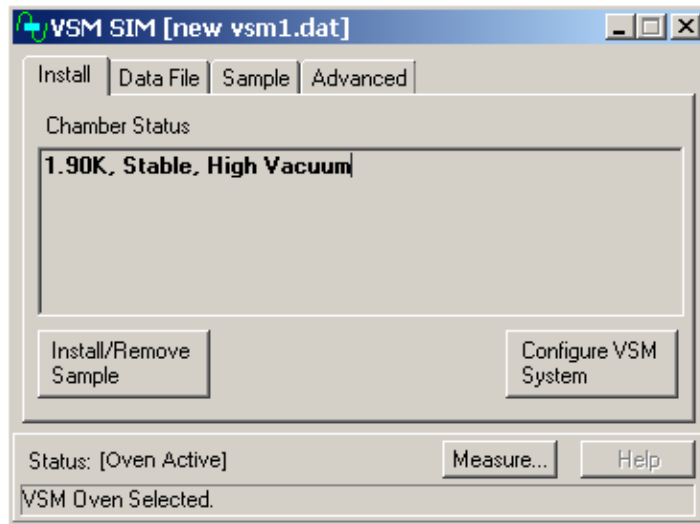


Figure 6-1. The VSM Control Center (oven enabled) after sample installation has been completed

6.4.2.1 STATUS AREA

The **Status** area at the bottom of the **VSM Control Center** includes new information such as the measurement mode you selected during the **VSM Install/Remove Sample Wizard**. For example, after you select VSM Oven mode and complete the **VSM Install/Remove Sample Wizard**, the **Status** area will report "Oven Active" and "VSM Oven Selected" (Figure 6-1). If you had selected standard VSM mode, the **Status** area would display "Standard VSM configuration selected."

6.4.2.2 THE VSM INSTALL/REMOVE SAMPLE WIZARD

The **VSM Install/Remove Sample Wizard** (**VSM Install** wizard for short) organizes the oven and sample-installation process through a set of automated dialog boxes. You start the **VSM Install** wizard by clicking on the **Install/Remove Sample** button located in the lower left of the **Install** tab (Figure 6-1). The initial pages of the **VSM Install** wizard for the VSM Oven-enabled software are somewhat different than those for the standard VSM software, most notably by including a dialog in which you choose to operate in Oven mode or standard VSM mode (Figure 6-2).

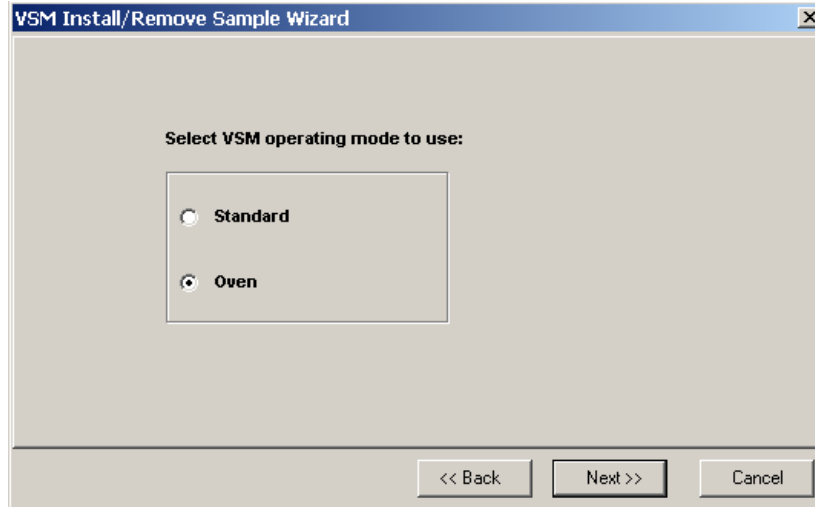


Figure 6-2. VSM Install wizard: Selecting the VSM operating mode

You can return to this page and change your operating mode during any part of the **VSM Install** wizard. However, if you want to change the operating mode after the **VSM Install** wizard has ended, you must restart the wizard from the beginning.

The **Sample Coordinates** pages for the two versions of the VSM software also differ. As shown in Figure 6-3, these pages display an oven heater stick instead of the VSM sample rod.

Note: A green dot appears in the illustration after a scan has been completed. The dot indicates the location of the sample on the heater stick.

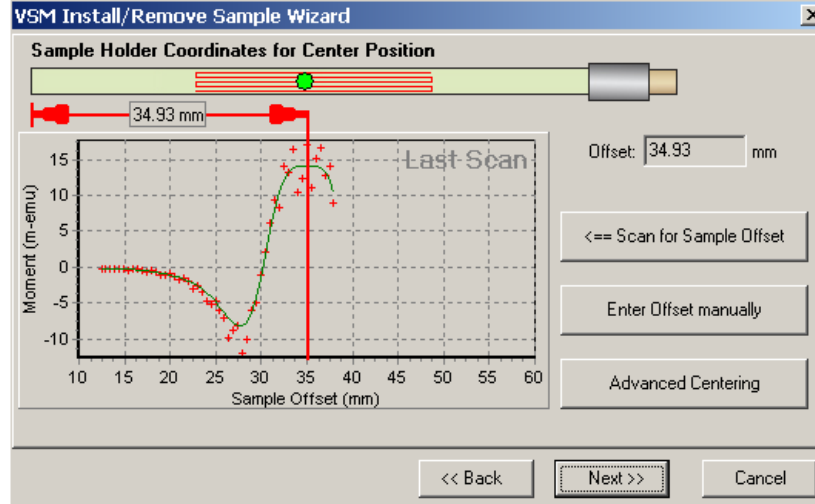


Figure 6-3. The Sample Coordinates page for determining sample offset

On the last page of the VSM Install wizard for the oven mode is a test for electrical continuity of the heater stick. Also, the system initiates high vacuum before completing the wizard.

Other than the above-mentioned changes to the **VSM Install** wizard, the same sample-installation process is used in the standard and oven modes.

6.4.3 VSM Oven Measurements

6.4.3.1 CENTERING TAB

While operating in VSM Oven mode, the normally recommended setting in the **Centering** tab is **No Automatic Centering**,¹ as is shown in Figure 6-4.

Because the thermal expansion of the 2.5 cm long heater region of the oven heater stick produces a negligible movement of the sample, touchdowns are typically unnecessary when you are using the oven mode.

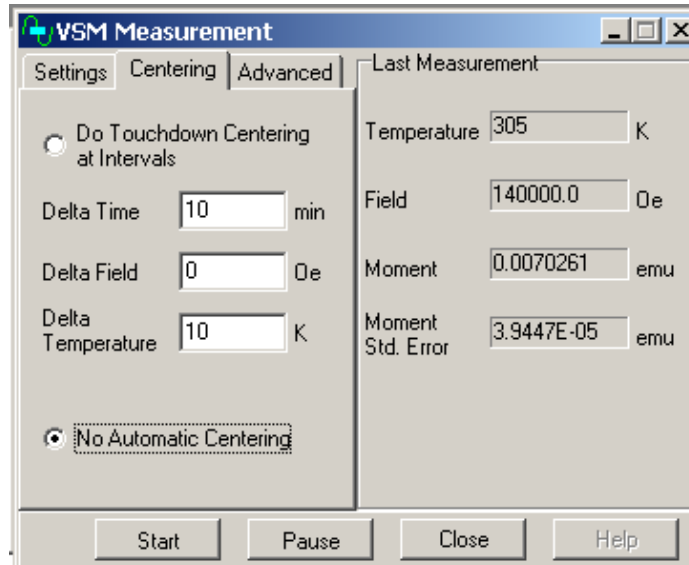


Figure 6-4. Centering tab of VSM Measurement dialog and default setting

Important: When you change to VSM Oven mode after operating at low temperatures in standard VSM mode, there will be a slight thermal expansion of the sample chamber. Hence, you will improve accuracy by using touchdowns for approximately 30 minutes after the sample reaches room temperature.

Refer to the *VSM Option User's Manual* for more information about touchdowns.

6.4.3.2 ADVANCED TAB

As is shown in the **Advanced** tab of the **Measure** dialog in Figure 6-5, the maximum VSM linear motor oscillation amplitude is 2 mm peak when the oven is operating, by comparison with 5 mm peak for the standard VSM. This difference is due to the increased heating of the linear motor drive coil when it is running in high vacuum instead of in the exchange gas atmosphere that is used for standard VSM measurements.

¹ Recall that **Do Touchdown Centering at Intervals** is the default in the standard VSM mode.

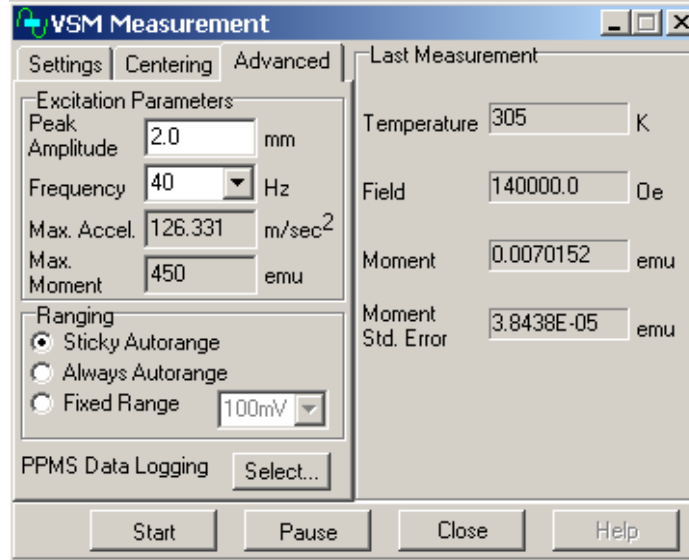


Figure 6-5. Advanced tab of VSM Measurement dialog box, showing peak amplitude during oven use

Model CM-C VSM Oven Module

A.1 Introduction

This appendix contains the following information:

- Section A.2 provides a functional overview of the Model CM-C VSM Oven module, including a block diagram and electrical specifications.
- Section A.3 describes the front panel and relevant components of the Model CM-C VSM Oven module.
- Section A.4 describes the rear panel and relevant components of the Model CM-C VSM Oven module.

A.2 Functional Overview

The Model CM-C VSM Oven module (4101-200) is a temperature-control module that was designed for the specific needs of the VSM Oven option. Figure A-1 shows the module and its front panel.

The principle function of this module is to provide the appropriate current to a platinum heater based on resistance feedback and temperature readings from a thermocouple and a thermistor. The maximum heater output is 25 V rms at either 50 Hz or 60 Hz with synchronous current readback detection. The temperature-control (PID) loop is closed digitally at about 10 Hz using a 16-bit current source. Other features include in-system programmable on-board flash memory for program storage and a serial ROM for calibration and other configuration data.

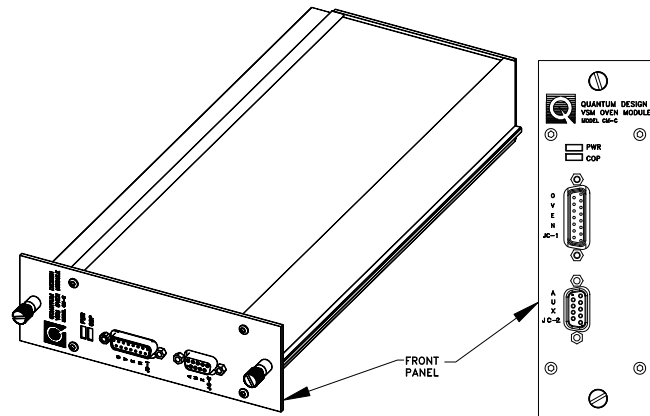


Figure A-1. Front panel of Model CM-C VSM Oven module

A.2.1 Functional Block Diagram

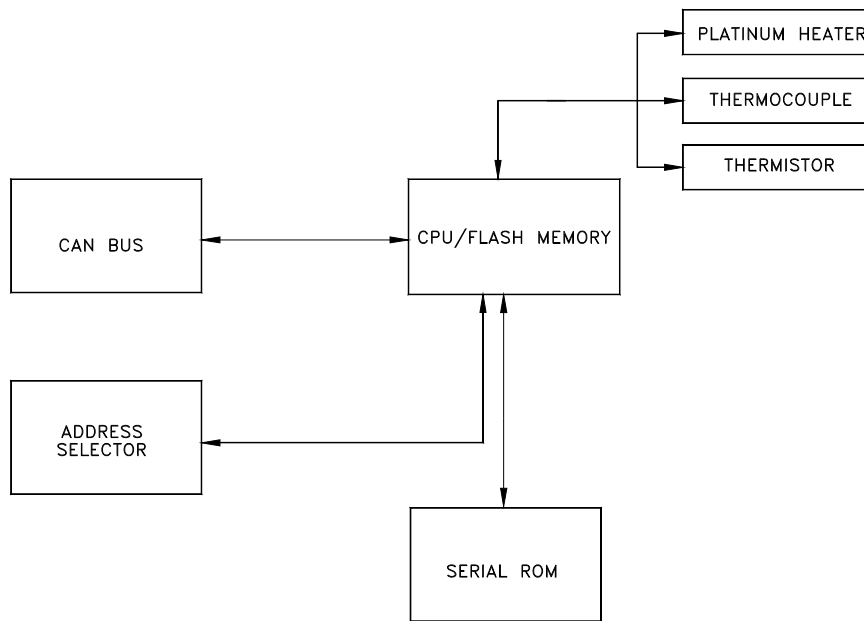


Figure A-2. Functional block diagram of Model CM-C VSM Oven module

A.2.2 Specifications

Table A-1. Electrical specifications for the Model CM-C VSM Oven module

Heater Voltage Compliance Limit	25 V rms
Supply Voltage	±24 V DC
Thermistor Resistance	10 k Ω at 25°C
Thermocouple Voltage (Max)	7.587 mV at 1000 K
Thermometer Sample Rate	14 Hz

A.3 Model CM-C VSM Oven Module: Front Panel

A.3.1 Indicator LEDs

The front panel of the Model CM-C VSM Oven module has two LEDs in the top left, as shown in Figure A-1. The PWR LED indicates the power-on status of the module. The COP (CANopen Protocol) LED indicates the status of the CAN network controller. Table A-2 outlines the LED states and provides solutions in the event of a problem.

Important: The error information in Table A-2 refers to situations that persist for longer than about 15 seconds. Typically, when the module is powered on, the LEDs might briefly flash red before they turn green. This is a normal part of the startup or reset sequence.

Table A-2. LED guide for Model CM-C VSM Oven module

LED	COLOR	STATUS	MEANING AND/OR SOLUTION
PWR	Green	On	The processor is running with no errors (normal).
	Red	Flashing	Errors were encountered during the self-test. The flashing sequence can be used to determine the cause of the failure.
COP	Green	On	CAN status is operational (normal).
		Flashing	CAN status is pre-operational. Verify that the cable is connected to PC.
	Red	On or flashing	Error on the CAN bus. Contact Quantum Design for assistance.

If you are unable to achieve operation with both LEDs green, please contact Quantum Design for assistance.

A.3.2 Front Panel Connectors and Pinout Tables

A.3.2.1 JC-1 OVEN CONNECTOR

This connector is used to supply current to the oven stick heater as well as to read temperature information from the thermocouple and the thermistor. Table A-3 shows the pinout connections from the oven-control cable to the oven module. Note that eight wires pass through to the JC-2 AUX connector (AUX1, AUX2, ..., AUX9).

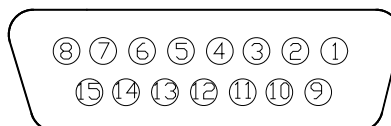


Figure A-3. JC-1 pin assignments on the Model CM-C VSM Oven module

Table A-3. JC-1 pinout connections from the oven-control cable to the Model CM-C VSM Oven module

PIN	FUNCTION
1	Thermocouple +
2	Thermistor A
3	AUX1
4	AUX2
5	AUX3
6	AUX4
7	Heater A
8	N/C
9	Thermocouple -
10	Thermistor B
11	AUX6
12	AUX7
13	AUX8
14	AUX9
15	Heater B

A.3.2.2 JC-2 AUX CONNECTOR

The JC-2 AUX connector is available for future expansion of the VSM Oven option, including transport measurements. Please contact Quantum Design before you attempt to use the connector. Note the pinout connections shown in Table A-3 for this connector.

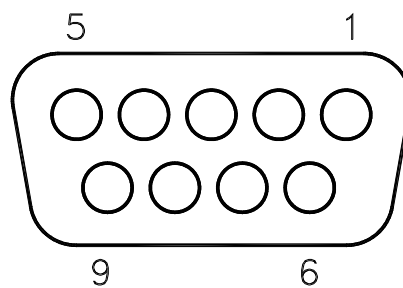


Figure A-4. JC-2 pin assignments on the AUX connector

A.4 Model CM-C VSM Oven Module: Rear Panel

The rear panel of the Model CM-C VSM Oven module contains an address selector, two guide holes, and the CAN connector through which the module sends and receives network data and receives power.

A.4.1.1 ADDRESS SELECTOR

Each module on the CAN bus must have a unique 5-bit binary address. The selector on the back panel is used to set the four least significant bits, while an internal jumper sets the most significant bit. If the selector is set to “0,” the module uses its default address. For a Model CM-C VSM Oven module, the default address is 12.

A.4.1.2 GUIDE HOLES

The two guide holes are used to align the connector with a low-power receptacle on the Model 1000 Modular Control System.

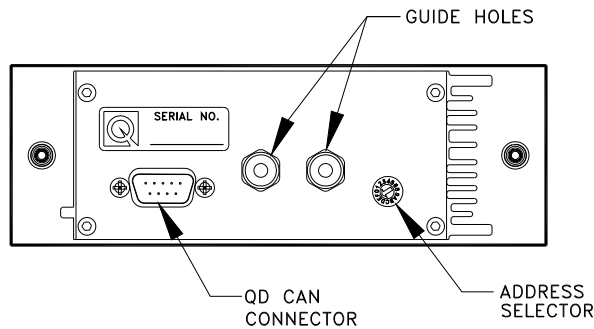


Figure A-5. Rear panel of Model CM-C VSM Oven module

A.4.1.3 QD CAN CONNECTOR AND PINOUT TABLE

The QD CAN connector is the main communication connection for controlling the Model CM-C VSM Oven module. The CAN network signals (CAN High, CAN Low) are connected to all other CAN modules on the bus and to the PC. Power (± 24 volts), reset, and sync signals also are sent to the module through this connector.

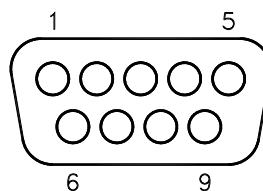


Figure A-6. QD CAN connector on rear of the Model CM-C VSM Oven module

Table A-4. Pinout table for QD CAN connector on rear of the Model CM-C VSM Oven module

PIN	FUNCTION
1	-24 V
2	CAN Low
3	Power Return (24V)
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High / Reset
9	+24 V DC

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



Electrical Transport Option (ETO) User's Manual

Part Number 1084-700, B1

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C O N T E N T S

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Contents and Conventions

P.1 Overview

The preface contains the following info:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 discusses the conventions of the manual.
- Section P.4 discusses the safety guidelines for this option.

P.2 Scope of the Manual

This manual discusses the operation of the Electrical Transport Option (ETO), option hardware, option software, and general use of the option.

This manual does not provide detailed information about the Versalab system or the MultiVu software application used to operate Versalab. This information can be found in the VersaLab User's Manual.

P.3 Conventions in the Manual

- | | |
|---------------------------|--|
| File menu | Bold text is used to distinguish the names of menus, options, buttons, and panels appearing on the computer screen. |
| File >> Open | The >> symbol indicates that you select multiple, nested software options. |
| <Enter> | Angle brackets distinguish the names of keys located on the PC keyboard. |
| Note | Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances. |



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



This symbol signals electrical hazards that could result in bodily harm, or loss of life. Used at all accessible 200-240 V power outlets.



This symbol signals information on fusing.

P.4 Safety Guidelines and Regulatory Information

Before using this product, please read the entire content of this manual and observe all instructions, warnings and cautions. These are provided to help you understand how to safely and properly use the Electrical Transport Option (ETO).

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 user serviceable by the user.

Theory of Operation

1.1 Introduction

This chapter contains the following information:

- Section 1.2 gives an overview of the Electrical Transport Option.
- Section 1.3 discusses the different measurements that are possible with ETO.

1.2 Overview of the Electrical Transport Option

The Quantum Design Electrical Transport Option (ETO) enables users to make several different types of transport measurements over a wide range of resistance values and sample types. The option contains two separate channels each with their own dedicated electronics to allow simultaneous, continuous resistance measurement of two different samples. Each channel's electronics consists of a precision current source and voltage preamplifiers coupled to a Digital Signal Processor (DSP). Measurements are usually made by applying a sinusoidal AC drive current and measuring the AC voltage response. However, a special 2-wire high resistance mode is available where an AC voltage is applied and the AC current response is measured with a current amplifier.

The DSP is used to filter the AC response and pick out the portion of the response at the same frequency and phase as the drive signal. All other components of the signal are filtered out, eliminating frequency dependent noise, DC offsets, and instrument drift. The DSP can also calculate harmonic components of the measured signal.

The current source has a minimum precision of 1 nA and a maximum current of 100 mA. It is capable of supplying both DC and AC current with frequencies from 0.1 Hz to 200 Hz. The preamplifiers consist of a high gain amplifier, a programmable gain amplifier, and a high impedance (current) amplifier. These three preamps, in conjunction with the current source, give the ETO a noise floor of 10 nΩ and allow measurement of resistances up to 5 GΩ.

The Electrical Transport option is available on both PPMS and VersaLab systems. The ETO software is integrated into the MultiVu software application used to control and monitor the system hardware. You may use any MultiVu commands while working with the ETO. This software integration allows you to utilize MultiVu sequences to fully automate system operations and run a wide range of measurements without being present in the laboratory.

1.3 Electrical Transport Option Measurement Types

The Electrical Transport Option supports three types of measurements:

- Resistance
- IV Curves
- Differential Resistance

1.3.1 Resistance

The ETO is capable of both four-terminal and two-terminal resistance measurements. In the classic four-terminal geometry (4-wire mode), two leads pass a current through the sample and two separate leads measure the potential drop across a section of the sample. Ideally the voltage leads draw very little or no current. The current through the sample and the potential drop across the sample can therefore be known to a high degree of accuracy. Ohm's law can then be used to calculate the resistance of the sample for the region between the two voltage leads. This geometry eliminates the effects of lead contact resistance from the measurement results.

The resistance is calculated using Ohm's law

$$R = \frac{V}{I} \quad (1)$$

where V is the measured potential drop across the sample and I is the current through the sample. Unlike the AC Transport measurement option available from Quantum Design, ETO does not calculate the resistivity. Since resistivity relies on sample specific parameters that may not be known, or not known accurately at measurement time, the option reports the resistance in units of Ohms. Resistivity can be calculated as follows,

$$\rho = \frac{RA}{\ell} \quad (2)$$

where R is the reported resistance, ℓ is the voltage lead separation, and A is the cross-sectional area through which the current is passed.

It is important to configure the leads correctly in order to take full advantage of the instrument's sensitivity. The current leads are typically located at the edge of the sample while the voltage leads lie between (Figure 1-1(a)). When current is passed through the sample an electric field is created. The voltage leads should be arranged such that they measure a potential drop across a region where the electric field lines are relatively straight. This is achieved by placing the voltage leads in-line with the current leads or by separating the voltage leads by a distance that is small compared to their distance from the current leads. It is also important for a *true* four-terminal measurement that the voltage and current leads do not contact the sample at the same point. Otherwise, the reported resistance will include the sample contact resistance.

The lead geometry shown in Figure 1-1(a) works well for thin film sample with well defined dimensions. Other lead geometries may be used depending on the nature of the sample. For instance, when measuring a bar-shaped sample you may attach the current leads to conductive pads that contact the entire end of the bar and then make voltage leads that

contact the sample in a line parallel to those ends. You are responsible for determining the best lead arrangement for your needs and for interpreting the resulting data.

The Electrical Transport option is also capable of measuring large resistances using a two-terminal lead geometry (2-wire mode). This mode is typically used for resistances above 10 MΩ. In 2-wire mode the system applies a voltage across the sample and uses the high impedance amplifier to measure the current passing through the sample. The resistance is again calculated using Ohms law. Figure 1-1(b) shows a sample wired up in the two-terminal configuration. To achieve the best results the sample should be rectangular or bar-shaped with the leads far apart yielding a well-defined measurement region where the electric field line density is uniform. Lead and contact resistance can usually be ignored in this case as long as they are much less than the sample resistance.

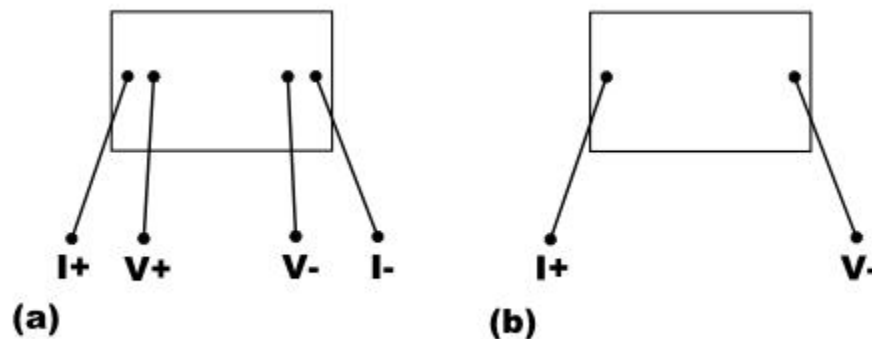


Figure 1-1. Typical lead placement on a rectangular thin-film sample for (a) four-wire and (b) two-wire lead geometries.

1.3.1.1 HARMONIC DETECTION

The second and third harmonics of the measured response are reported in units of dB during a resistance measurement. The units of dB are referenced to the voltage of the fundamental response. This information can be related to nonlinearities in the sample but is often an indicator of the amount of noise present during a measurement. The second and third harmonic contribution is typically less than -50 dB for a good measurement.

1.3.1.2 HALL COEFFICIENT

While it is not explicitly supported it is possible, with proper lead placement, to perform 4-wire Hall measurements using the Electrical Transport Option. When charged particles move perpendicular to a magnetic field a force is exerted on them perpendicular to both the field and the direction of particle motion. The force can be expressed as

$$\vec{F} = q\vec{v} \times \vec{B} \quad (3)$$

This transverse force can often cause charge carriers to build up on one edge of a sample leading to a potential difference across the sample. This potential difference is called the Hall potential, V_H . The sign of the Hall potential generally indicates the sign of the charge carriers and the

magnitude of the potential is related to the density of charge carriers in the sample. The Hall coefficient, R_H , describes these two properties and is defined as,

$$R_H = \frac{E_H}{jB} = \frac{V_H A}{I \ell B} \quad (4)$$

where E_H is the Hall field, j is the current density, and B is the magnitude of the magnetic field. For a well defined geometry the current density is equal to I/A where I is the current and A is the sample cross-sectional area, and the Hall field is equal to V_H/ℓ . Here ℓ is the distance between a set of transverse voltage leads used to measure the Hall potential. It can be shown that the $R_H = (nq)^{-1}$, with n representing the number of charge carriers per unit volume in the sample and q representing the charge of the carriers.

To measure the Hall potential arrange the voltage leads perpendicular to both the current and field directions. Figure 1-2 shows a thin-film rectangular sample wired up to detect the Hall potential. In this example the magnetic field would be applied normal to the sample surface. Using Eq. (1) you can rewrite Eq. (4) in terms of the resistance, R , reported by ETO.

$$R_H = \frac{RA}{\ell B} \quad (5)$$

Accurately measuring the Hall potential can be difficult. To measure the potential difference due only to the Hall potential the voltage leads must be perfectly perpendicular to the bias field. If this is not the case the measured potential will include a component due to the longitudinal potential resulting from the sample resistance. The magnitude of the longitudinal potential is typically much larger than the Hall potential making even small errors in lead placement problematic. Both the Hall potential and the longitudinal potential are proportional to the bias current so the AC filtering technique employed by the ETO does not eliminate this effect.

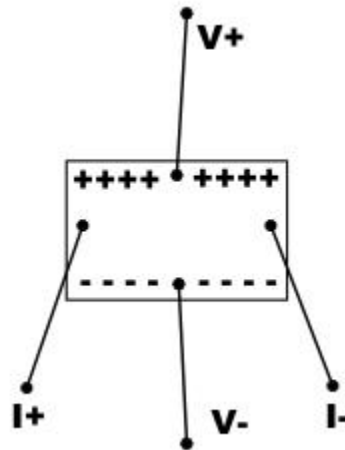


Figure 1-2. Typical lead placement on a rectangular thin-film sample for measurement of the Hall potential. The magnetic field would be applied normal to the sample surface.

1.3.2 Current-Voltage Curves

The ETO is capable of measuring the current-voltage characteristics of any sample or device. In this mode the electronics applies a triangular excitation waveform and measures a response waveform. These two waveforms are combined to form the current-voltage curve for the device under test. The triangular excitation waveform always starts and ends at zero bias. Figure 1-3(a) shows both an excitation waveform and the response waveform for a diode plotted as a function of time. The nonlinear device characteristics are clearly visible in the response waveform. Figure 1-3(b) shows resulting I-V curve when the current waveform is plotted as a function of the voltage waveform. The ETO software allows you to select the number of quadrants to measure and in what order. The resulting curve will contain 256 points per quadrant. Both four-wire and two-wire IV curves can be measured. In the four-wire case a current excitation waveform is used and a voltage response waveform measured. In the two-wire case a voltage excitation waveform is used and a current response waveform measured.

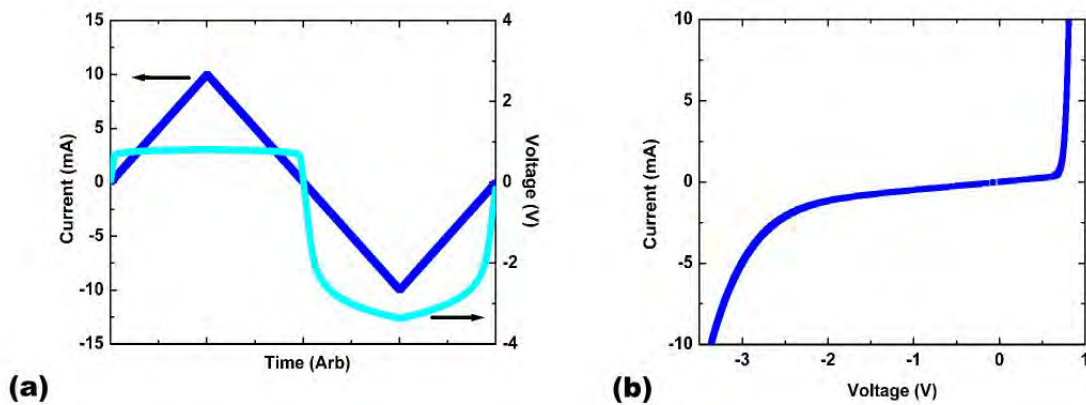


Figure 1-3. Two graphs showing current and voltage waveforms (a) and the resulting I-V curve (b) for a diode measured using the ETO.

1.3.3 Differential Resistance Curves

The ETO is capable of measuring differential resistance as a function of bias current or voltage. This is achieved by applying a small AC excitation on top of a DC offset bias. The AC response is measured and used to calculate the differential resistance in the same manner as the standard resistance measurement when no DC bias signal is applied. The differential resistance is a direct measure of the first derivative of the IV curve at a given DC bias. This type of measurement is very useful for examining small nonlinearities in a given device.

Installation

2.1 Introduction

This chapter contains the following information:

- Section 2.2 lists the components of the Electrical Transport Option and describes the procedure for installation on PPMs and VersaLab systems.

2.2 Initial Installation of Hardware and Software

This section describes the procedures for the initial installation of the Quantum Design Electrical Transport Option (ETO) onto the PPMS and VersaLab systems. These procedures apply only to the first time you install and use ETO. Normal activation and use of the option is covered in Chapter 4.

Note: If your system came from Quantum Design with ETO already installed you do not need to follow the procedures in this chapter. Refer to Chapter 4 for normal operation.

Table 2-1 lists the components of the Quantum Design Electrical Transport Option. Please verify that you have received all the components before you begin the installation process.

Table 2-1. A list of hardware components comprising the Electrical Transport Option for (a) PPMS, (b) VersaLab, and (c) DynaCool systems.

(a)	PPMS COMPONENT	PART NUMBER
	ETO Module CM-H	4101-451
	ETO Head EM-QN	4101-455
	ETO Module Cable	3101-455-02
	ETO Sample Cable	3101-456-01
	ETO Fan Cable	3101-457-02
	ETO Accessory Kit	4084-710

(b)

VERSALAB COMPONENT	PART NUMBER
ETO Module CM-H	4101-451
ETO Head EM-QN	4101-455
Accessory Mounting Kit	4372-079
ETO Module Cable	3101-455-01
ETO Sample Cable	3101-456-01
ETO Fan Cable	3101-457-01
ETO Accessory Kit	4084-710

(c)

DYNACOOOL COMPONENT	PART NUMBER
ETO Module CM-H	4101-451
ETO Head EM-QN	4101-455
ETO Module Cable	3101-455-01
ETO Sample Cable	3101-456-01
ETO Fan Cable	3101-457-01
ETO Accessory Kit	4084-710

2.2.1 Install the ETO Module (4101-451) and ETO Head (4101-455)

Use this section only if the ETO electronics have not been installed on your PPMS or VersaLab system.

2.2.1.1 PPMS HARDWARE INSTALLATION

Refer to the PPMS User's Manual for any issues regarding the PPMS. These installation instructions assume the Model 1000 tower and CAN bus have already been installed and tested.

1. Turn off the power to the Model 1000 CAN tower.
2. Verify that the power is off. All of the module LEDs should be dark.
3. Remove the cover plate for one of the rear, high-power, module bays and carefully insert the module into the bay. Gently finger-tighten the two mounting screws to secure the module in place. The module should now be flush with the other modules or cover plates.
4. The ETO head needs to be mounted close to the Grey Lemo connector at the back of the sample chamber. Determine the optimal mounting location for the ETO head and place the two pieces of self-adhesive grip tape on the PPMS dewar at this location. Make sure the head is close enough that the sample cable can easily reach the Grey Lemo without stressing the cable. Refer section 3.3 (cables and jumpers) for the recommended ETO head mounting location.
5. Press the ETO head onto the grip tape to secure it in place. Make sure the head is not touching any other dewar components.
6. Connect the ETO Module Cable (3101-455-02) and Fan Cable (3101-457-02) between the ETO module and the ETO head.

7. Turn on the power to the Model 6000 tower. Verify that both the PWR and COP indicator LEDs on the module are green. It may take up to 30 seconds for the module to boot and the LEDs to turn green. The COP LED will blink green until the option is activated.

2.2.1.2 VERSALAB HARDWARE INSTALLATION

Refer to the VersaLab User's Manual for any issues regarding the VersaLab system.

1. Turn off the power to the VersaLab system.
2. Open the hood covering the CAN rack and verify that none of the modules already installed have power. All of the module LEDs should be dark.
3. Install the Accessory Mounting Bracket to the VersaLab frame using the two #6-32 screws, washers, and nuts provided in the Accessory Mounting Kit.
4. Remove the cover plate for the left most module bay as viewed when facing VersaLab. Carefully insert the module into the bay. Gently finger-tighten the two mounting screws to secure the module in place. The module should now be flush with the other modules or cover plates.
5. Mount the ETO Head to the accessory-mounting bracket using the hook and loop fastening tape on both the head and the bracket. Be sure to mount the head in the correct orientation. Refer section 3.3 (cables and jumpers) for the recommended ETO head mounting location.

Note: When installed properly, the ETO head should not touch any of the mounting screws and should not interfere with the hood when closed. Adjust the location of the head if necessary.

6. Connect the ETO Module Cable (3101-455-01) and Fan Cable (3101-457-01) between the ETO module and the ETO head.
7. Turn on the power to the VersaLab system. Verify that both the PWR and COP indicator LEDs on the module are green. It may take up to 30 seconds for the module to boot and the LEDs to turn green. The COP LED will blink green until the option is activated.

2.2.1.3 DYNACOOOL HARDWARE INSTALLATION

1. Turn off the power to the PPMS DynaCool CAN rack located on the right side of the cryostat. The power switch for the CAN rack is located in back.
2. Open the CAN rack door and verify that none of the modules installed have power. All of the module LEDs should be dark.
3. Install the two pieces of Velcro provided with the system on the back panel of the CAN rack between the CAN rack and the cryostat shield. These should be in the center of the back panel high enough to ensure the ETO sample cable can reach the grey lemo connector. Refer section 3.3 (cables and jumpers) for the recommended ETO head mounting location.
4. Remove the cover for the second or third module bay from the back and carefully insert the ETO module. Finger tighten the two mounting screws to secure the module in place. The module should now be flush with the other modules or cover plates.
5. Mount the ETO head on the Velcro between the CAN rack and the cryostat with the D-shell connector facing the back of the PPMS DynaCool.
6. Connect the ETO Module Cable (3101-455-01) and Fan Cable (3101-457-01) between the ETO module and the ETO head. Attach the ETO Sample Cable (3101-456-01) between the ETO head and the grey lemo connector.

7. Turn on the power to the CAN rack. Verify that both the PWR and COP indicator LEDs on the module are green. It may take up to 30 seconds for the module to boot and the LEDs to turn green. The COP LED will blink green until the option is activated.

2.2.2 Install the ETO Software

Use the following instructions to install the VersaLab Electrical Transport Option software on your PC. (See the MultiVu User's Manual for MultiVu installation instructions.) The installation wizard will install all of the option files in the C:\QDPPMS\ETOption\, C:\QDVersaLab\ETOption\, or C:\QDDynacool\ETOption directory for PPMS and VersaLab respectively.

1. Close all other applications prior to installing the ETO software.
2. Double click the ETO Installer.msi file to start the installation wizard. Follow the instructions on the screen to install the software.
Note: Be sure to select the correct base system type, either PPMS or VersaLab, when prompted.
3. Verify that the ETO software is installed correctly by activating it within the MultiVu software.
 - a. Open the MultiVu application software.
 - b. Select **Utilities>>Activate Option**.
 - c. In the **Option Manager** dialog box select **Electrical Transport** from the **Available Options** list and press the **Activate** button.
 - d. **Electrical Transport** will move to the **Active Options** list inside the **Option Manager**.
 - e. The Electrical Transport software will open the **ETO Console** window and the **ETO Event Log** window, confirming that the software was install correctly.

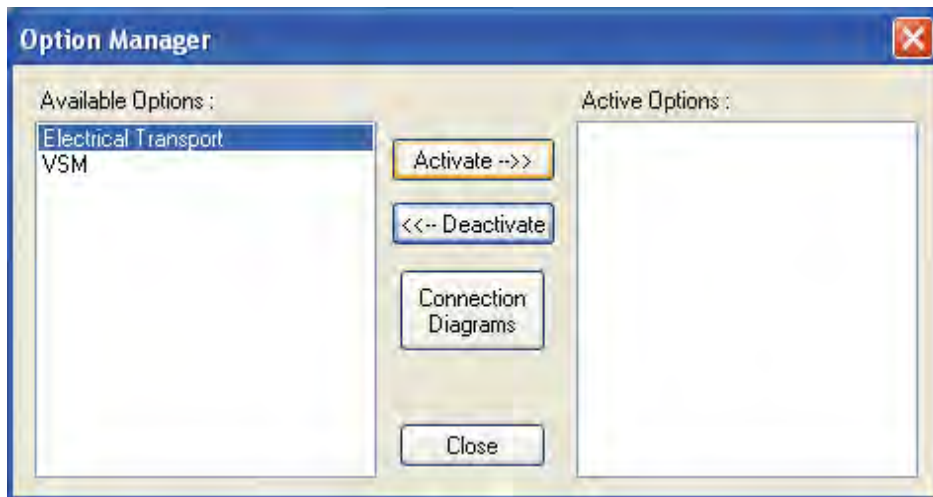


Figure 2-1. Activate the Electrical Transport Option using the Option Manager dialog box.

2.2.3 Configuring the Electronics

The ETO electronics are ready to use out of the box and do not require any calibration files on the PC. All of the calibration and configuration information is stored inside the module SROM. Contact your customer service representative with any problems associated with the calibration or SROM.

Hardware

3.1 Introduction

This chapter contains the following information:

- Section 3.2 discusses the operation of the ETO electronics.
- Section 3.3 discusses the cables and jumpers.
- Section 3.4 discusses the sample pucks.

3.2 ETO Module and Head

The Electrical Transport Option electronics consists of the module (CM-H) and the remotely mounted head (EM-QN). The module contains a Digital Signal Processor (DSP) that performs all AC stimulus-response calculations and a secondary processor for post processing and communications. The remotely mounted head contains two sets of current drivers and preamplifiers to allow for simultaneous, independent resistance measurements on channel 1 and channel 2. Figure 3-1 shows the module and head enclosures.

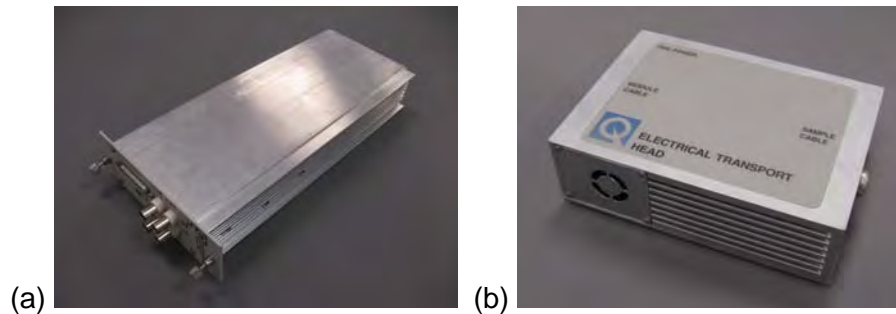


Figure 3-1. Pictures of (a) the ETO module (CM-H) and (b) the remotely mounted ETO head (EM-QN)

To perform a basic transport measurement an AC drive signal is generated at the requested frequency by the DSP. The current driver board in the head receives the drive signal and converts it to a current that is applied to the sample. The preamplifier board in the head detects and amplifies the sample response signal and sends it back to the DSP for processing. The DSP calculates the in-phase and out of phase components of the response signal as well as several harmonic amplitudes.

The module (CM-H) contains four BNC outputs for use in monitoring the drive (JH-2 and JH-4) and response signals (JH-1 and JH-3) for both channel 1 and 2. The BNC labels are located on the module front panel as shown in Figure 3-2. The drive signal full-scale is 10 V. For example, on the 100 mA range, a 50 mA peak excitation current would generate a 5 V peak signal at the BNC. The response signal is monitored behind the preamplifiers. The response signal measured at the BNC needs to be divided by the total gain to obtain the voltage at the sample.



Figure 3-2. Picture of the ETO module front panel showing the CH1 1 measured signal BNC (JH-1), CH1 drive signal BNC (JH-2), CH2 measured signal BNC (JH-3), CH2 drive signal BNC (JH-4), the head cable connector (JH-5), and the fan cable connector (JH-6).

3.2.1 Current Driver Electronics

The current drivers in ETO employ active feedback to approximate an ideal current source. When the feedback is on, the current driver adjusts the applied potential to maintain a constant output current regardless of the sample and lead resistance. Figure 3-1 (b) shows the load lines for the current driver with feedback on. As the graph shows, the current is independent of the measured voltage across the sample. The driver behaves as a current source for potentials up to the 30 V compliance of the source. In this mode the current driver has high output impedance. This is the default operating mode and is desirable for most measurements and samples.

When feedback is off, the drivers can be represented as a voltage source behind a source resistance. Figure 3-3 (a) shows the equivalent circuit when the feedback is turned off. In this case, the current passing through the sample is dependent on the sample and lead resistance. Figure 3-3 (c) shows the load lines for the current driver as a function of the measured sample voltage. In this configuration the sample current decreases with increasing sample voltage. If the sample or lead impedance is comparable or larger than the source impedance the actual current will be significantly lower than the requested current. ETO will correct for this discrepancy as best as possible and the corrected current will be reported in the data file. The corrected current will be a good approximation of the actual sample current in the limit that $R_{Leads} \ll R_{Drive}$. For systems with large lead resistance the current can be manually corrected as follows:

$$I_{sample} = \frac{I_{reported} \cdot R_{Drive}}{R_{Drive} + R_{Leads}}$$

where R_{Leads} is the resistance due to leads and sample contacts. Table 3-1 lists the nominal drive impedance for each current range when the drive feedback is disabled.

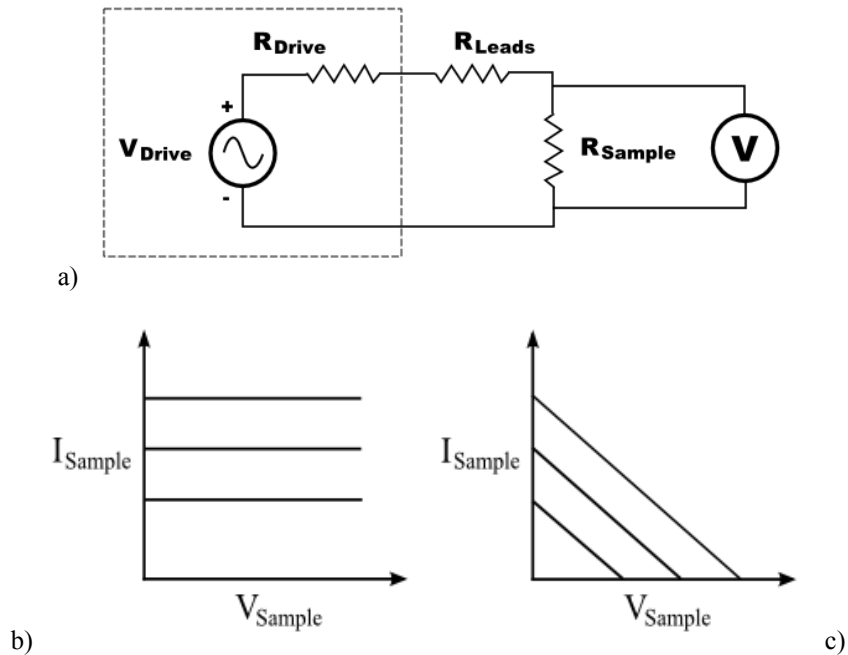


Figure 3-3. The equivalent circuit for ETO when drive feedback is disabled. The dashed line represents the current source equivalent circuit.

Table 3-1. ETO current drive output impedance with feedback disabled.

Current Range	Drive Output Impedance
100 mA	100 Ω
10 mA	1 k Ω
1 mA	10 k Ω
100 μ A	100 k Ω
10 μ A	1 M Ω
1 μ A	10 M Ω
100 nA	100 M Ω

In most cases the electronics should be run with the drive feedback enabled. This will typically yield the most accurate measurement. However, it may be useful to disabled the feedback if you are measuring an IV curve on a sample with a non-linear current-voltage characteristic, such as a diode. With feedback off the IV curve of a diode will contain more points at lower bias currents when the device impedance is large.

3.2.2 Preamplifiers

The ETO head contains a programmable-gain amplifier (PGA), a high-gain amplifier (HGA), and a high-impedance amplifier (HZA). The amplifiers used in a particular measurement are determined both by the measurement type and the range specified in the measurement window. Four-wire measurements will use the PGA and the HGA when needed while two-wire measurements will use the PGA and the HZA at all times. The HGA has a typical noise specification of $1 \text{ nV} / \sqrt{\text{Hz}}$ giving a noise floor of 10 nΩ for a 100 mA excitation. The HZA, by contrast, is a sensitive amp meter and is used to measure impedances above 10 MΩ.

The gain of the preamplifiers is determined in each measurement by selecting the range in the preamp controls. The range is typically specified as a voltage. This can be interpreted as the maximum sample voltage the system can measure at the chosen range. The lower the voltage range the higher the total gain. In practice the system can measure up to 110% of the voltage specified by the range. If you do not know the impedance of your sample ahead of time, you can elect to autorange. In this case the system will automatically adjust the range based on the measured signal. Selecting autorange will delay the return of data while the system adjusts the gains to find the appropriate range.

Table 3-2. Preamp ranges and their associated gains for both 4-wire and 2-wire measurement modes.

4-Wire Mode			
Preamp Nominal Range	Module Gain	Head Gain	Total Gain
4 V	1X	1X	1X
1.3 V	3X	1X	3X
0.4 V	1X	10X	10X
130 mV	3X	10X	30X
40 mV	1X	100X	100X
13 mV	1X	300X	300X
4 mV	3X	300X	900X
1.3 mV	1X	3,000X	3,000X
0.4 mV	3X	3,000X	9,000X
130 uV	1X	30,000X	30,000X
40 uV	3X	30,000X	90,000X
2-Wire Mode			
Preamp Nominal Range	Module Gain	Head Gain	Total Gain
400 nA	1X	1X	1X
130 nA	3X	1X	3X
40 nA	1X	10X	10X
13 nA	3X	10X	30X
4 nA	1X	100X	100X
1.3 nA	3X	100X	300X

If you are measuring a sample with impedance greater than 10 M Ω you will use the HZA and a 2-wire sample-mounting configuration. In this configuration ETO applies a voltage and measures the current through the sample. The preamp range is specified in Amps for these measurements. The measurement windows will automatically switch the drop down list to a selection of current ranges when the 2-wire configuration is chosen.

3.3 Cables and Jumpers

The ETO option includes one 25-pin cable to connect the module to the remotely mounted head, one 2-pin cable to power the cooling fans on the head, and one 8-pin cable to connect the head to the sample chamber. The cable use and routing are shown in Figure 3-4. ETO kits intended for use on PPMSs are shipped with long length module and fan cables. ETO kits intended for use on VersaLab systems are shipped with short length cables. Both kits use the same sample cable to connect the head to the sample chamber grey Lemo. Additional sample cables are necessary when connecting ETO to one of the temperature options available on the PPMS.

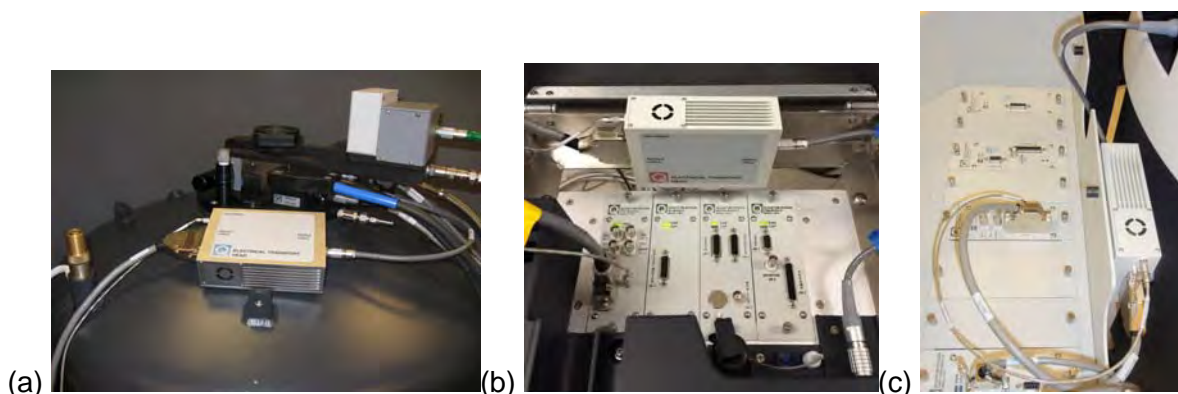


Figure 3-4. Pictures of the ETO option installed onto (a) a PPMS system (b) a VersaLab system and (c) Dynacool system. On the PPMS the head is mounted on the dewar using grip tape. VersaLab requires the use of the accessory mounting kit (4372-079).

3.4 Sample Pucks

ETO includes two sample pucks for mounting samples that interface with the VersaLab sample chamber wiring. The pucks have a mounted circuit board with labeled contact pads for convenient sample wiring on 3 channels. When using ETO, only channels 1 and 2 are used. Channel 3 may be used with other measurement options or other third-party electronics that you hook up to the base system. The gold plated contact pads on the puck are useful for either wire bonding or soldering. For more information regarding sample wiring refer to Section 5.2.

Software

4.1 Introduction

This chapter contains the following information:

- Section 4.2 presents an overview of the ETO software including the ETO console.
- Section 4.3 reviews how to create data files and contains descriptions of each data column contained within the file.

4.2 Overview of Electrical Transport Option Software

4.2.1 ETO Console

Whenever the Electrical Transport Option is active the **ETO Console** window will be present within the MultiVu window. This window can be minimized but will only close when the option is deactivated. From the console window you can perform all basic system operations, such as sample installation, setting up data files, and running measurements manually in immediate mode. The **ETO Console** contains a **Setup** tab, **Data File** tab, and a **Measurement** tab. The functionality of each tab is described below.

4.2.1.1 SETUP TAB

The **Setup** tab is the default tab for the **ETO Console** and is displayed when the option is initially activated. Figure 4-1 shows the **Setup** tab within the **ETO Console**. This tab is used to install or remove samples and enter information about the samples that will be entered into the data file.

The **Sample Installation Wizard** button will launch a separate window for the wizard when pressed. This wizard will guide you through the steps necessary to open the sample chamber to either install or remove a sample. Figure 4-2 shows the **Sample Installation Wizard** window. The **System Status** panel displays the current system status and the **Instructions** panel displays information and guidance regarding each step of the wizard. The wizard does not execute any

commands when started and requires that the user press the **Open Chamber** button to initiate the installation procedure. Follow the instructions on the screen to install or remove a new sample.

The **Sample Properties** button will launch a separate window to allow you to enter properties of the sample that you would like to store in the data file. This information is stored in the data file header. For more information regarding the ETO data files refer to Section 4.3. Figure 4-3 shows the **Sample Properties** window containing the **Wiring**, **Sample 1 Info**, and **Sample 2 Info** tabs. The **Wiring** tab is used to specify the wiring configuration used on each sample. It also lists the types of measurements that can be run with each configuration. The two **Sample Info** tabs allow you to enter sample specific information such as name, material, and dimensions. For more information regarding the wiring configuration refer to Section 5.2.

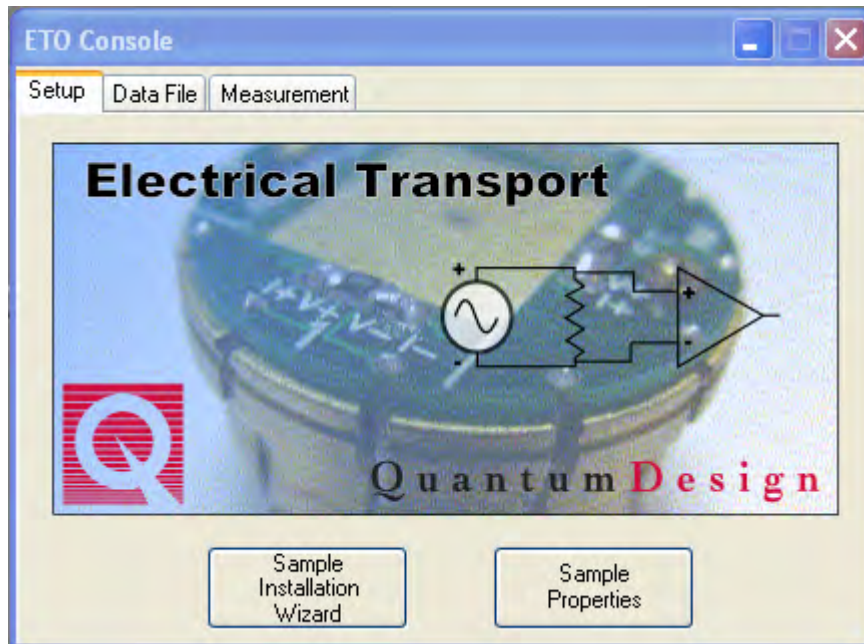


Figure 4-1. Setup tab in the ETO Console window.



Figure 4-2. The Sample Installation Wizard window.

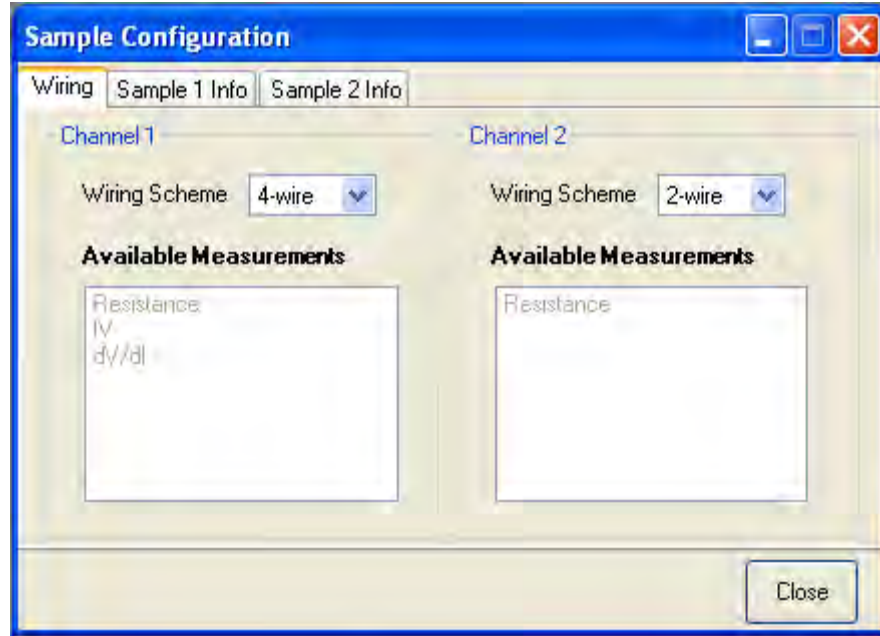


Figure 4-3. The Sample Configuration window.

4.2.1.2 DATA FILE TAB

The **Data File** tab is used to specify the data file into which the ETO data is written. Pressing the **Browse** button will bring up a standard Windows file interface that allows you to specify a file name and location on the PC for the data file. You can use this window to either select an existing file or create a new file name. If a new data file name is selected the data file is not created immediately. Instead the file is created when the first data point is written to the file. The currently selected target file and its location are displayed in the **Path** and **File Name** fields. The **Create new file version** checkbox, when checked, will add a numeric suffix to the file name displayed if the file already exists. If this box is unchecked the option will append to an existing data file.

The **Sample Properties** button is also contained in the **Data File** tab in the event the **Setup** tab is not used. The functionality for this button is identical to that on the **Setup** tab and you can enter information from either tab. The information contained in the **Sample Properties** window is written to the data file when it is first created. New sample information can be entered at any time before a new measurement is taken and the new file is created.

The **View** button opens the selected data file and displays the data as a new graph in MultiVu. It will always open the file specified in the **File Name** field provided the file has already been created.



Figure 4-4. Data File tab in the ETO Console window.

4.2.1.3 MEASUREMENT TAB

The **Measurement** tab is used to open the measurement window for the selected measurement. The radio buttons on the right indicate the measurement type selected and the display window gives an example of the type of data that can be collected. After you have selected the measurement type press the **Launch Measurement** button to bring up the immediate mode measurement window. While an immediate mode measurement window is active this tab is not active. The active measurement window must be closed prior to launching a different measurement window.

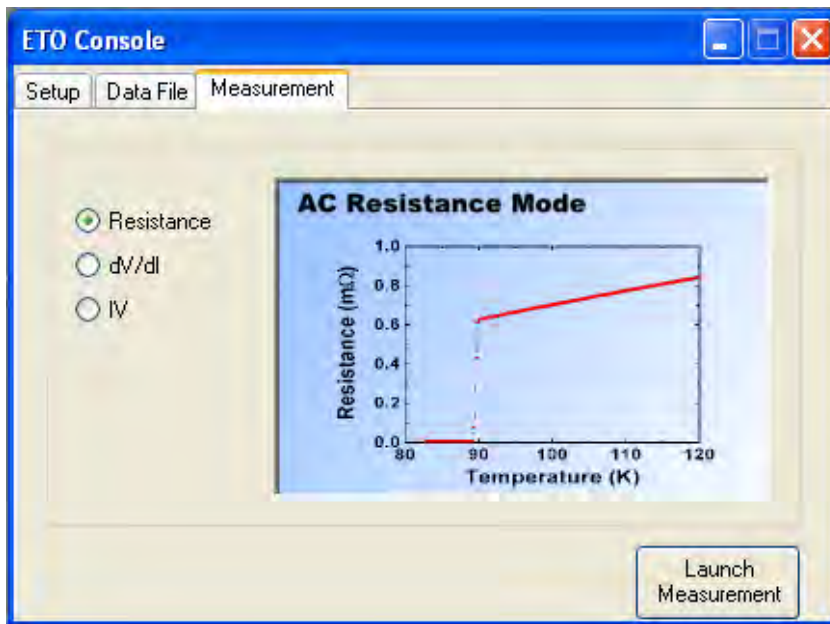


Figure 4-5. Measurement tab in the ETO Console window.

4.2.2 Electrical Transport Option Event Log

The **ETO Event Log** window is displayed when ETO is activated. This window displays high-level hardware and software activity during ETO measurements. This log will also display warnings, errors, and informational messages generated since option activation. If you have a problem while a measurement is running, entries in the event log can help diagnose the problem.

4.3 Electrical Transport Option Data Files

All of the data taken by the Electrical Transport Option is stored in a single data file. The file name and location can be set on the **Data File** tab of the **ETO Console** or within a sequence. For more information on changing the data file refer to Section 4.2.1.2. This section contains a list and description of each of the data columns as well as the measurement types that use the different data types.

Table 4-1. ETO Data Files

Data Type	Units	Description	Resistance	dV/dI	IV
Time Stamp	s	Time stamp associated with the row of data	X	X	X
Temperature	K	Sample Temperature	X	X	X
Field	Oe	Magnetic Field	X	X	X

Sample Position	deg	Angle of the sample stage relative to the magnetic field	X	X	X
Chamber Pressure	Torr	Pressure inside the sample chamber	X	X	X
Resistance Ch1/Ch2	Ohms	Measure resistance	X	X	
Resistance Std. Dev. Ch1/Ch2	Ohms	The standard deviation of the resistance	X	X	
Phase Angel Ch1/Ch2	deg		X	X	
I-V Current Ch1/Ch2	mA	Wave form of the current			X
I-V Current Ch1/Ch2	V	Wave form of the voltage			X
Frequency Ch1/Ch2	Hz	Excitation Frequency	X	X	X
Averaging Time Ch1/Ch2	s	Actual averaging time	X	X	X
AC Current Ch1/Ch2	mA	The peak amplitude of the AC drive excitation	X	X	X
DC Current Ch1/Ch2	mA	The DC drive excitation			X
Voltage Ampl. (Ch1/Ch2)	V	Magnitude of the voltage	X	X	X
In phase Voltage Ampl. (Ch1/Ch2)	V	The in-phase voltage amplitude	X	X	
Quadrature Voltage	V	The out-of-phase voltage amplitude	X	X	
AC Voltage Ch1/Ch2	V	The peak voltage of the AC drive	X	X	X
DC Voltage Ch1/Ch2	V	The maximum DC voltage in the channel	X	X	X
Current Ampl. (Ch1/Ch2)	mA	Magnitude of the current	X	X	X
In Phase Current Ampl. (Ch1/Ch2)	V	The in-phase current amplitude	X	X	
Quadrature Current Ch1/Ch2	mA	The out-of-phase current amplitude	X	X	
Gain	1-90000	Total gain = Module gain X head gain	X	X	X
2 nd Harmonic	V	The in-phase voltage amplitude of the 2 nd harmonic	X	X	
3 rd Harmonic	V	The in-phase voltage amplitude of the 3 rd harmonic	X	X	
ETO Status Code	N	An encoded integer containing information on the current status of the ETO hardware	X	X	X
ETO Measurement Mode	V	Integer indicating the measurement type for the row of data	X	X	X

ETO Measurements

5.1 Introduction

This chapter contains the following information:


- Section 5.2 discusses how to mount samples on the sample pucks.
- Section 5.3 discusses how to make measurements in immediate mode and in sequence mode.

5.2 Sample Mounting

The Electrical Transport Option includes two resistivity pucks that mount into the sample chamber. Each sample puck has a circuit board around the perimeter with 12 pads organized into three channels. Each channel has 4 pads labeled I+, I-, V+, V- in the standard 4 wire resistance geometry. The circuit board has labels for each pad as well as the channel number corresponding to each group to avoid confusion during sample mounting. ETO uses the channel 1 and channel 2 pads. There is a space in the center of the puck for physically mounting your sample substrate onto the puck surface. For most measurements below room temperature Apeazon N-grease is adequate to secure your sample to the puck surface. However, more exotic mounting methods may be necessary for larger samples.

The wiring configuration used for each sample depends on the type of sample you are measuring. The ETO electronics has a 4-wire standard mode for samples whose resistance is below 10 M Ω and a 2-wire high-impedance mode for samples whose resistance is 10 M Ω up to 5 G Ω . Both wiring configurations are discussed below along with mounting and wiring examples. Since the ETO has two independent sets of measurement electronics it is possible to wire up one sample in standard mode and the other in high-impedance mode.

Note: How you wire up your sample determines what electronics you must use. You cannot switch back and forth between standard and high-impedance modes without physically changing the sample wiring.



WARNING!

Do not wire up both channels to the same sample as this may lead to errors in the reported resistance due to stray capacitance.

5.2.1 4-Wire Mounting Configuration

To make a standard resistance measurement in 4-wire configuration you should mount your sample as shown in Figure 5-1(a) for the sampled wired to channel 2. The two current leads should be at the ends of your sample and the two voltage leads should be in line between the current leads. Ensure that the polarity of the voltage leads matches that of the current leads or the reported resistance will be negative.

5.2.1.1 HALL COEFFICIENT MOUNTING CONFIGURATION

ETO can also be used to make Hall measurements. Figure 5-1(b) shows channel 1 wired for a standard resistance measurement and channel 2 wired for a Hall measurement. The wiring arrangement shown yields the proper sign for the Hall coefficient. It is important when wiring a Hall sample that the voltage contacts are at the same zero-field potential to avoid measuring longitudinal signal. This is usually achieved by making sure the voltage contacts are perpendicular to the current contacts. ETO does not support 5-wire Hall measurements.

5.2.2 2-Wire Mounting Configuration

For samples whose resistance exceeds 10 M Ω the 2-wire configuration is necessary. In this case the sample is connected to the I+ pad and the V- pad as shown in Figure 5-1(a) channel 1. It is important that you use the I+ and V- pad or the measured resistance will have the wrong sign or value. Hall measurements are not possible in the 2-wire configuration.

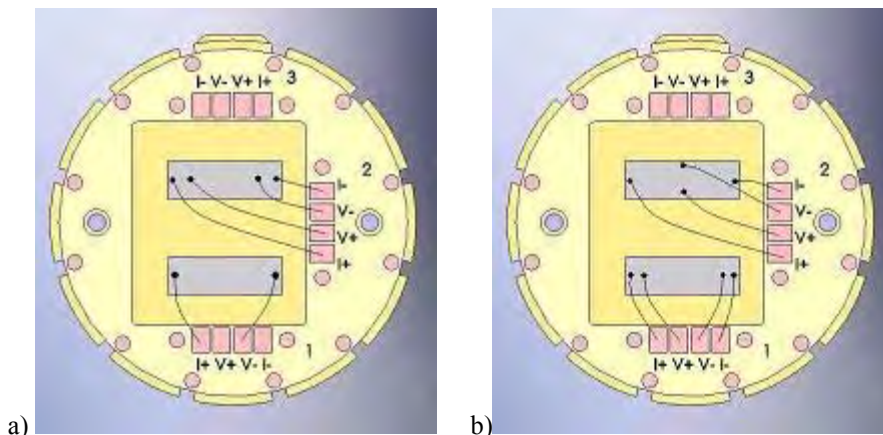


Figure 5-1. Sample wiring examples. (a) Shows mounted both a 2-wire sample on channel 1 and a 4 wire sample on channel 1. (b) Shows mounted a 4-wire sample on channel 1 and a 4-wire Hall sample on channel 2.

5.3 Sample Installation

The sample installation wizard is accessible from the **Setup** tab on the **ETO Console**. You are encouraged to use this wizard as it automates the installation process and leaves the sample chamber in the proper state for temperature cycling. Pressing the **Sample Installation Wizard** button will launch the wizard window, shown in Figure 4-2. Follow the instructions on the screen to install your sample. When prompted by the wizard install the sample puck using the puck extraction tool. (For more information on sample installation refer to either the PPMS or Versalab manual.) Pressing cancel at any time during the wizard will abort the wizard safely.

Enter sample information after installing the sample puck into the sample chamber. Press the **Sample Properties** button to bring up the dialog window. Enter the wiring configuration for sample 1 and 2 on the wiring tab. Enter any other sample specific information you desire on the two sample tabs. This information will be entered into the header section of the data file.

Switch to the **Data File** tab to select a new data file for your measurements. Press **Browse** to enter a new file name and location. Checking the *Create New File Version* checkbox will add a numeric suffix to the end of the file name if the file already exists. Press the **View** button to open the data file in MultiVu. **Note:** The data file is not created until a measurement is initiated.

5.4 Immediate Mode Measurements

After the sample puck has been loaded you are ready to take measurements in immediate mode. At this point it is a good idea to make a few test measurements in immediate mode to ensure your sample is loaded properly. Open up a measurement window to perform the desired test measurements. The immediate mode measurement windows are accessible through the **Measurement** tab in the **ETO Console**. Select a measurement type and press the **Launch Measurement** button. The following sections describe the process of setting up each type of measurement.

5.4.1 Taking Resistance Measurements in Immediate Mode

The AC Resistance Measurement window contains two identical sets of controls and indicators corresponding to channel 1 and channel 2. Use these blocks to define the measurement parameters and read the measurement results specific to each channel. A third section at the bottom of the window displays base-system information relevant to both channels. Figure 5-2 shows the resistance measurement window.

To make a measurement enable one or both channels, set all of the measurement parameters, then press the **Measure** button at the bottom. Resistance measurements will run until both channels are finished. Make sure to check the *Save Results to File* checkbox if you want to save the data. Measurement results will be displayed in the bottom of the window. Take the following steps to run an immediate mode measurement.

1. Check the **Enable Measurement** checkbox corresponding to the channel(s) you would like to measure.
2. Select either 4-wire or 2-wire from the **Sample Wiring** drop down menu corresponding to the wiring configuration used when the sample was mounted.

3. Use the **Amplitude** text box to specify the peak AC amplitude of the sinusoidal excitation applied during the measurement. Make sure the excitation is not large enough to damage your sample. The **Amplitude** will have units of mA for 4-wire configurations and in V for 2-wire.
4. Select the excitation frequency from the **Freq** drop down menu.
5. Select the **Range** from the drop down menu or check the **Autorange** check box if you want the range to be determined for you. The **Range** will have units of volts for 4-wire configurations and amps for 2-wire.
6. Use the **Averaging Time** text box to specify the measurement time in seconds.
7. Use the **Number of Measurements** text box to specify the number of resistance points you would like the measurement to include.
8. Press the **Measure** button to start the measurement.

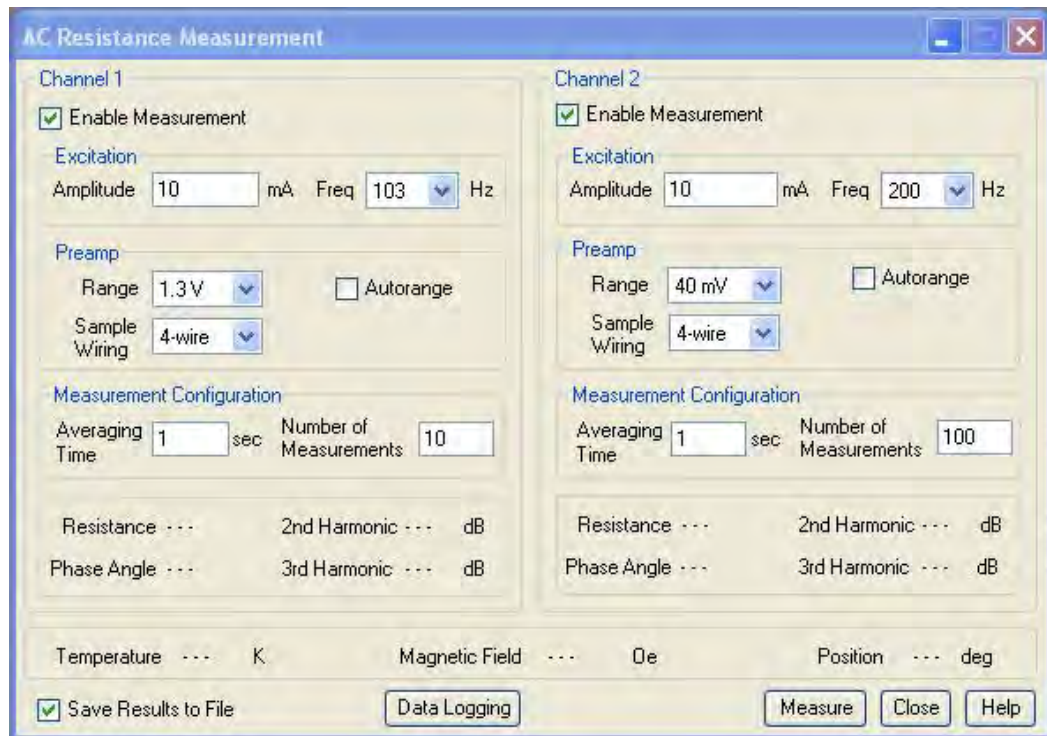


Figure 5-2. The immediate mode AC Resistance Measurement Window.

During the measurement the **Measure** button will read **Stop**. Press this button at any time to end the measurement. The measurement results will be displayed at the bottom of the window. The **Resistance** displays the resistance measured in Ω . The **Phase Angle** displays the phase angle in degrees between the excitation signal and the measured signal. The **2nd Harmonic** and **3rd Harmonic** display the amplitude of each harmonic in dB. At the very bottom of the window are displayed the **Temperature**, **Magnetic Field**, and **Position** of the samples inside the sample chamber.

5.4.2 Current-Voltage Curves

Current-voltage curves can be taken on only one channel at a time. Take the following steps to measure a current-voltage curve in immediate mode.

1. Select the radio button for **Sample 1** or **Sample 2** to select a channel.
2. Select either 4-wire or 2-wire configuration from the **Sample Wiring** drop down list.
3. Select the sweep frequency from the **Frequency** drop down list.
4. Use the **AC Amplitude** text box to specify the peak amplitude of the sweep. The AC Amplitude has the units mA with 4-wire configuration and V in 2-wire.
5. Make sure the **Disable Drive Feedback** box is un-checked. This feedback should only be disabled if you are measuring a sample with a highly non-linear I-V characteristic. See section XXXX for more details regarding the drive feedback.
6. Select the type of excitation waveform desired. You have the option of performing a four-quadrant sweep or a two-quadrant sweep in the positive or negative direction.
7. Use the **IV Sweeps** text box to specify the number of sweeps to average together to create the IV curve.
8. Select the **Range** from the drop down menu.
9. Press the **Measure** button to begin the measurement.

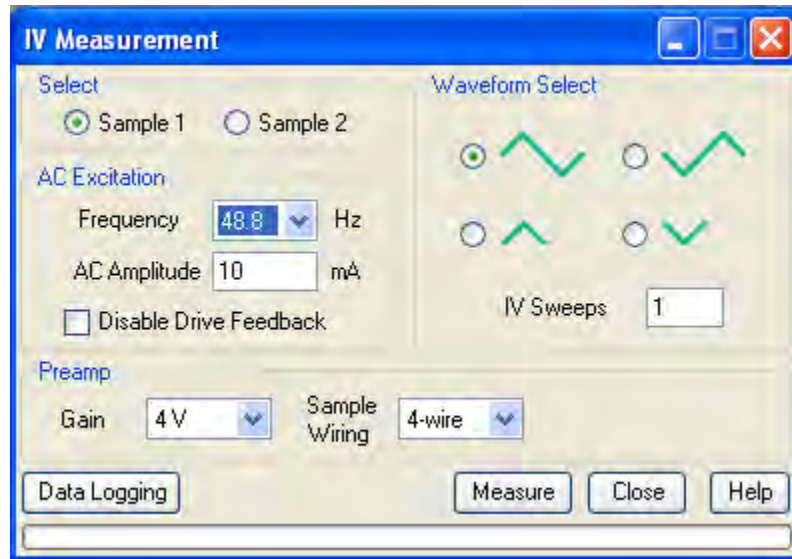


Figure 5-3. The immediate mode IV measurement window.

While the measurement is being performed the **Measure** button will display **Stop**. Press this at any time to abort the current measurement. The status bar at the bottom of the window will display the measurement progress. When the status bar is full the IV curve will be available in the data file.

5.4.3 Differential Resistance Curves

Differential resistance curves, like IV curves, can only be performed on one channel at a time. This measurement applies a small AC excitation and measures the differential resistance at various DC bias points. Take the following steps to measure a differential resistance curve in immediate mode.

1. Select the radio button for **Sample 1** or **Sample 2** to select a channel.
2. Select either 4-wire or 2-wire configuration from the **Sample Wiring drop** down list.
3. Select the AC frequency from the **Frequency** drop down list.
4. Use the **DC Amplitude** text box to specify the maximum value of the DC sweep.
5. Use the **Steps/Quadrant** text box to specify the number of DC steps per quadrant of the DC sweep.
6. The **Step Size** will be filled in automatically using the previously entered information. If you prefer, you can enter the step size and the steps per quadrant will be recalculated.
7. Select the DC sweep quadrants by dragging the start and stop cursors around on the sweep graph.
8. If the sweep does not start at zero bias, select the **Ramp to Starting Point** check box to ramp the DC bias to the starting value prior to starting the measurement.
9. Use the **AC Amplitude** text box to specify the amplitude of the AC excitation used to measure the differential resistance. This value should be less than the DC sweep amplitude.
10. Select the **Range** from the drop down menu.
11. Use the **Averaging Time** text box to specify how long to average each data point.
12. Use the **Settling Time** text box to specify how long in milliseconds to wait after each DC step before beginning each new resistance measurement. 100 ms is usually adequate.
13. Press the **Measure** button to begin measuring the curve.

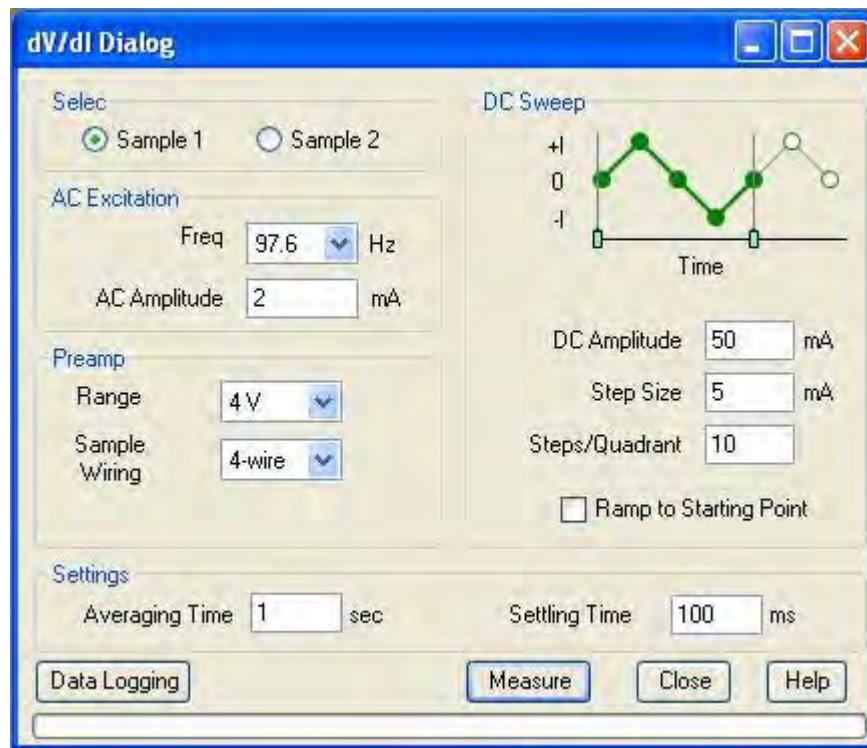


Figure 5-4. The immediate mode differential resistance measurement window.

While the measurement is being performed the **Measure** button will display **Stop**. Press this at any time to abort the current differential resistance measurement. The status bar at the bottom of the window will display the measurement progress. When the status bar is full the dV/dI curve will be available in the data file.

5.5 Sequence Measurements

A sequence can be used to run a series of measurements sequentially or to change other system properties while measuring, such as temperature or magnetic field. Any number of measurement commands or system commands can be run from within a sequence. Figure 5-5 shows the list of sequence commands available in MultiVu when ETO is active. All ETO specific commands are listed under *Measurement Commands >>Electrical Transport*.

Note: For detailed information about creating and editing sequence files and for a discussion about all standard sequence commands, refer to the PPMS or VersaLab User's Manual.

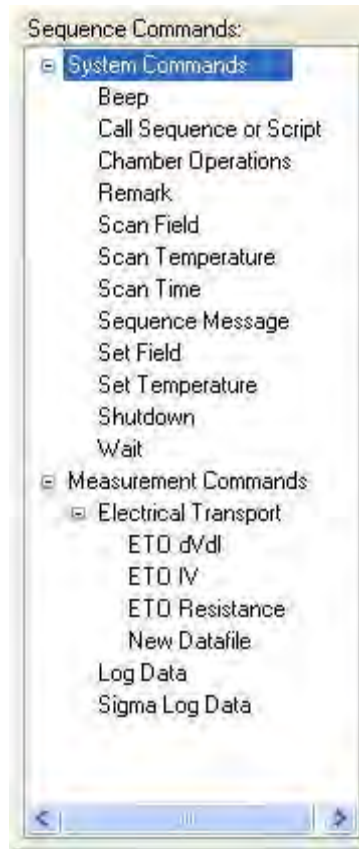


Figure 5-5. List of sequence commands available when ETO is active.

5.5.1 ETO Resistance

Figure 5-6 shows the sequence command window for the resistance measurement. This window contains the same controls as the immediate mode window. Follow the same steps outlined in Section 5.4.1 to specify the measurement parameters. When you are finished press the OK button and the measurement command will appear in the sequence window.

Since this command allows simultaneous measurements on both channel 1 and 2 it is possible for the two channels to have different measurement times. This can be caused by either very different averaging times or by selecting different numbers of points to measure on each channel. In this case the sequence will wait for both channels to finish measuring before moving on to the next sequence command.

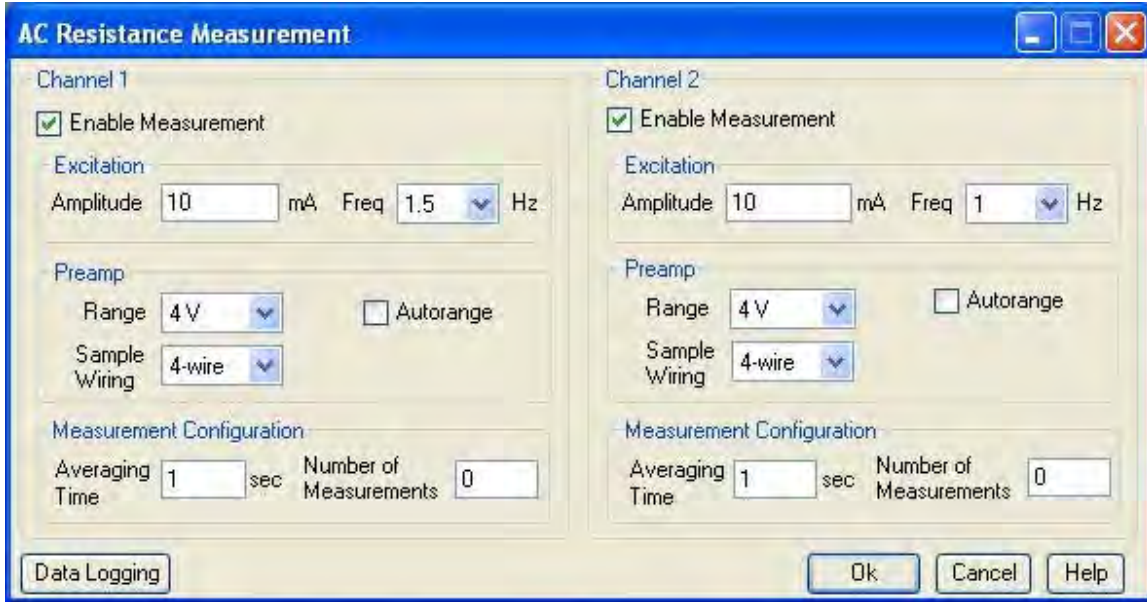


Figure 5-6. The sequence command window for resistance measurements.

5.5.2 ETO dV/dI

Figure 5-7 shows the sequence command window for differential resistance measurements. Follow the same steps outlined in Section 5.4.3 to specify the measurement parameters. When you are finished, press the OK button and the measurement command will appear in the sequence window. As with the immediate mode, the sequence command for differential resistance can only perform measurements on one channel at a time. To measure both channels requires two sequence commands.

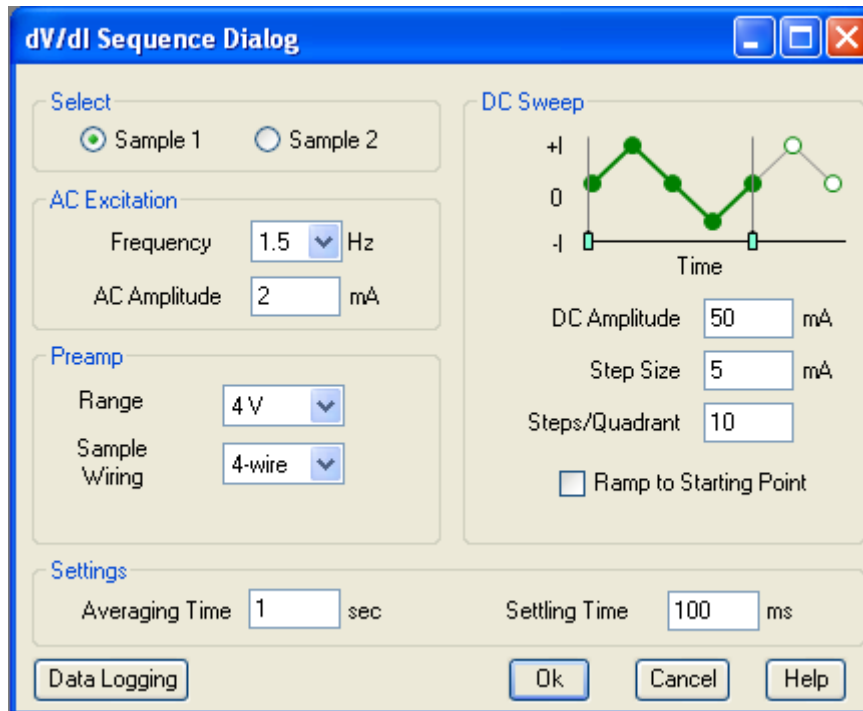


Figure 5-7. The sequence command window for differential resistance measurements.

5.5.3 ETO IV

Figure 5-8 shows the sequence command window for the IV curve measurements. Follow the same steps outlined in Section 5.4.2 to specify the measurement parameters. When you are finished press the OK button and the measurement command will appear in the sequence window. As with the immediate mode the sequence command for IV curves can only perform measurements on one channel at a time. To measure both channels requires two sequence commands.

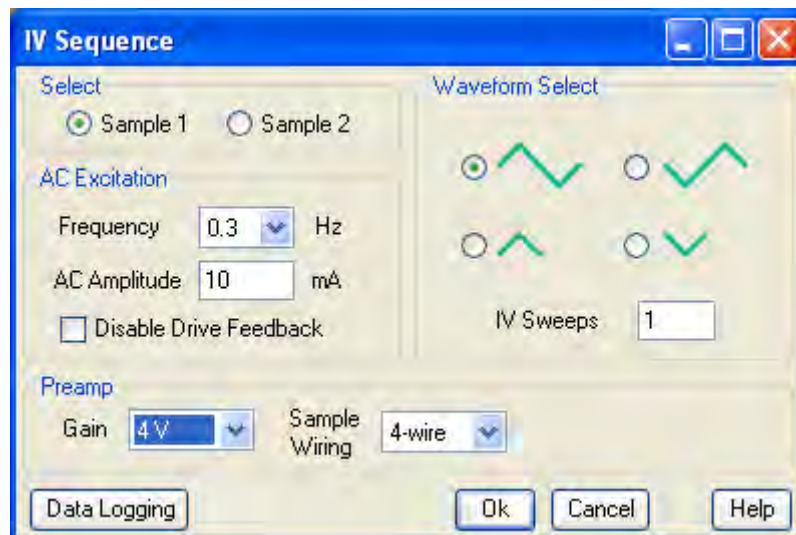


Figure 5-8. The sequence command window for IV measurements.

5.5.4 New Data File

Figure 5-9 shows the sequence command window to select or create a new data file within a sequence. Insert this command whenever you need to switch files, such as between measurements of different types. Press the **Browse** button to open the file dialog and select the file. Selecting the **Create New File/Version** radio button will create a new file with the name specified or add a numeric suffix if the file already exists. Selecting the **Append to File** radio button will add the data to an existing data file.

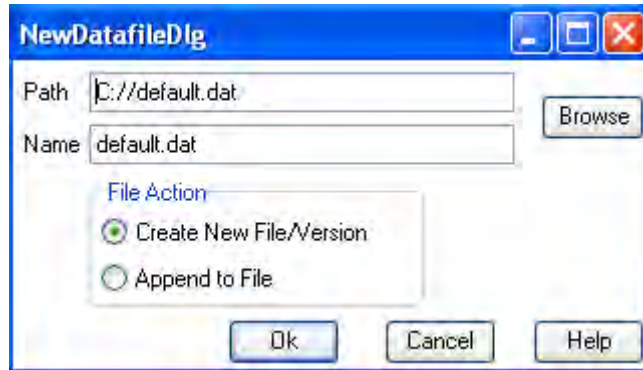


Figure 5-9. The sequence command window for creating a New Data File.

Operation with the Horizontal Rotator

6.1 Introduction

This chapter contains the following information:

- Section 6.2 presents an overview of ETO operation with the Horizontal Rotator.
- Section 6.3 explains how to configure the rotator thermometer.
- Section 6.4 discusses how to take ETO measurements when using the Horizontal Rotator.
- Section 6.5 contains interconnection tables for the ETO/Horizontal Rotator probe cable.

6.2 Overview of ETO Operation with the Horizontal Rotator

Many researchers find it useful to be able to rotate samples in the magnetic field of the PPMS sample chamber while measuring with the ETO system. This can be accomplished by using the PPMS Horizontal Rotator in conjunction with the ETO option. However, there are some important considerations when the two options are used together to take measurements. This chapter explains those important considerations. When the ETO electronics is used with the Horizontal Rotator, a Rotator/MFP Experiment cable (part number 3084-010-01 and -02) is also required for PPMS and Dynacool PPMS (or Versalab) respectively.

When the ETO system uses the Horizontal Rotator probe, four of the sample chamber wires must be connected directly to the rotator thermometer. Only eight wires—rather than the normal twelve—are available for ETO measurements. This cable (see Figure 6-1) splits off the four thermometer wires that go to the system bridge board from the dedicated Rotator wiring leads where the sample is connected.

Refer to the *Physical Property Measurement System: Horizontal Rotator Option User's Manual* for more information about using the Horizontal Rotator.

6.3 Rotator Thermometer Configuration

6.3.1 In PPMS with Model 6000

The rotator thermometer can be used only if it is plugged into the user bridge board or the system bridge board in the Model 6000 PPMS Controller. The Rotator/MFP experiment cable shipped with the Horizontal Rotator allows the rotator thermometer signals to be directed to the bridge board while sample signals are directed to the ETO electronics.

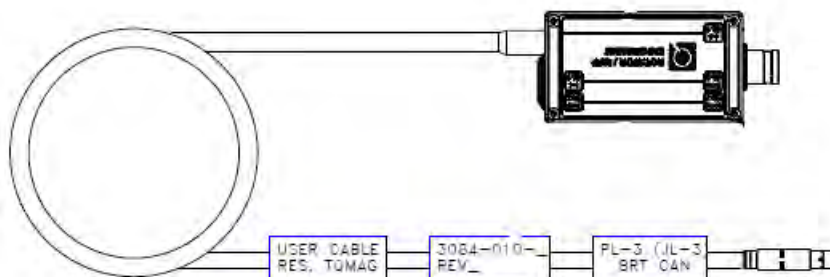


Figure 6-1. Rotator/MFP Experiment Cable

When the system is using the Rotator/MFP Experiment cable (part number 3084-010-01), the rotator thermometer should be connected to the system bridge board (P2) in the Model 6000. The following steps need to be completed to configure the PPMS and use the rotator thermometer:

1. Vent the PPMS sample chamber and install the rotator/motor assembly (part number 4084-303-01 for low or 02 for high resolution). Then insert the rotator probe (part number 4084-304) into the sample chamber.
2. If you can find the **PPMS 32-bit Tools** icon on the PC desktop, go to the next step. Otherwise, insert the disk included with the Horizontal Rotator option into the PC, and select the D: drive. Install PPMS 32-bit Tools in the C: drive and create an icon on the PC desktop.
3. Select the **PPMS 32-bit Tools** icon on the PC desktop, and then run the Romcfg32 utility.
4. Select **Send to PPMS | Send Config** in the main menu.
5. Select the C: drive, which is the drive where the rotator configuration files are located. If you cannot find them, select the D: drive, which contains copies of the rotator configuration files.
6. Select the HRETO###.cfg file, where ### represents the serial number for your rotator. This uses the Sys bridge channel 4 and maps that source (code 42) to map channel 23 where we read out the rotator temperature. The format of the configuration file is as follows:

```
Movecfg 1,0.0532,380,0; (or 1,0.00449,380,0; for high resolution)
$sysbrdg 4,1000,100,0,0,10;
Mapdat 23,42;
TABLE 23,2,2,#0 (Temperature Calibration Table Here)
TblMode 23,1;
```

```
Usertemp 23,1.8,1.7 2 1.00;
```

The HRETO###.cfg file directs the thermometer calibration data to the system bridge board (4 pin Lemo connector on P2 Port) in the Model 6000. Note that the HR###.cfg file directs the thermometer calibration data to the user bridge board for use with the Resistivity option and the standard user bridge cable supplied with that option. The *Physical Property Measurement System: Horizontal Rotator Option User's Manual* explains how you configure the user bridge board to read the rotator thermometer.

Both HRETO###.cfg and HR###.cfg also configure the motor for proper step sizes according to which type of motor you are using. (Configuration files for high resolution motors have “-H” at the end of the file name.)

7. Select **OK**.
8. Install the Rotator/MFP experiment cable by plugging the connectors on the cable into the ports indicated by the cable labels. Refer to Figure 6-1.
 - Plug the 14-pin gray Lemo connector on the Rotator/MFP experiment cable into the gray, color-coded port on the probe head.
 - Plug the 4-pin Lemo connector on the Rotator/MFP experiment cable labeled “P2 System Bridge” into the small round port next to the “P2–System Bridge” port on the rear of the Model 6000.
 - Plug the 14-pin gray Lemo connector on the ETO sample cable (part number 3101-456-01) into the 14-pin gray Lemo socket on the Rotator/MFP experiment cable box. Plug the 8-pin Lemo connector of the ETO module cable to the ETO Head assembly (part number 4101-455).
 - Connect ETO Module cable (part number 3101-455-02) and fan cable (part number 3101-457-02) between the ETO Head assembly and the ETO module (part number 4101-451).

The PPMS now uses the rotator thermometer to control the temperature and the rotator motor is configured properly. Data files written by the ETO software now report the rotator thermometer reading, in K, under the item labeled “PPMS Map 23.” For Rotator options shipped prior to March 2012, a simple modification to the HRACT###.cfg file is required to allow the ETO software to report the rotator temperature. Quantum Design Customer Service can help you with this file modification.

If you connect the Rotator/MFP experiment cable to the probe head while the Horizontal Rotator probe is installed, you are essentially connecting a current source to a calibrated, negative-temperature-coefficient, resistive thermometer with resistance between 20–10,000 Ω .

6.3.1.1 TURNING OFF USERTEMP

Configuring the Horizontal Rotator by transmitting the configuration files to the Model 6000 ROM also turns on the User Temp so that the rotator thermometer is used for temperature control. When you complete your experiments with the Horizontal Rotator and remove the Horizontal Rotator probe from the sample chamber, you should turn off this thermometer. Complete the following steps:

1. Open **Monitor QD-6000** in the **PPMS 32-bit Tools** folder, or select **Utilities>>Send GPIB Commands** in the PPMS MultiVu interface.
2. Type `USERTEMP 0`.
3. Press <Enter> to execute this command.

If you do turn off the User Temp, the resistance read off the Block thermometer is used to control the temperature in the sample chamber. If the User Temp remains on, but no user thermometer is installed—that is, the Horizontal Rotator probe is removed from the PPMS—the temperature control defaults back to the Block thermometer, but returns to the User Temp whenever a resistance is measured there.

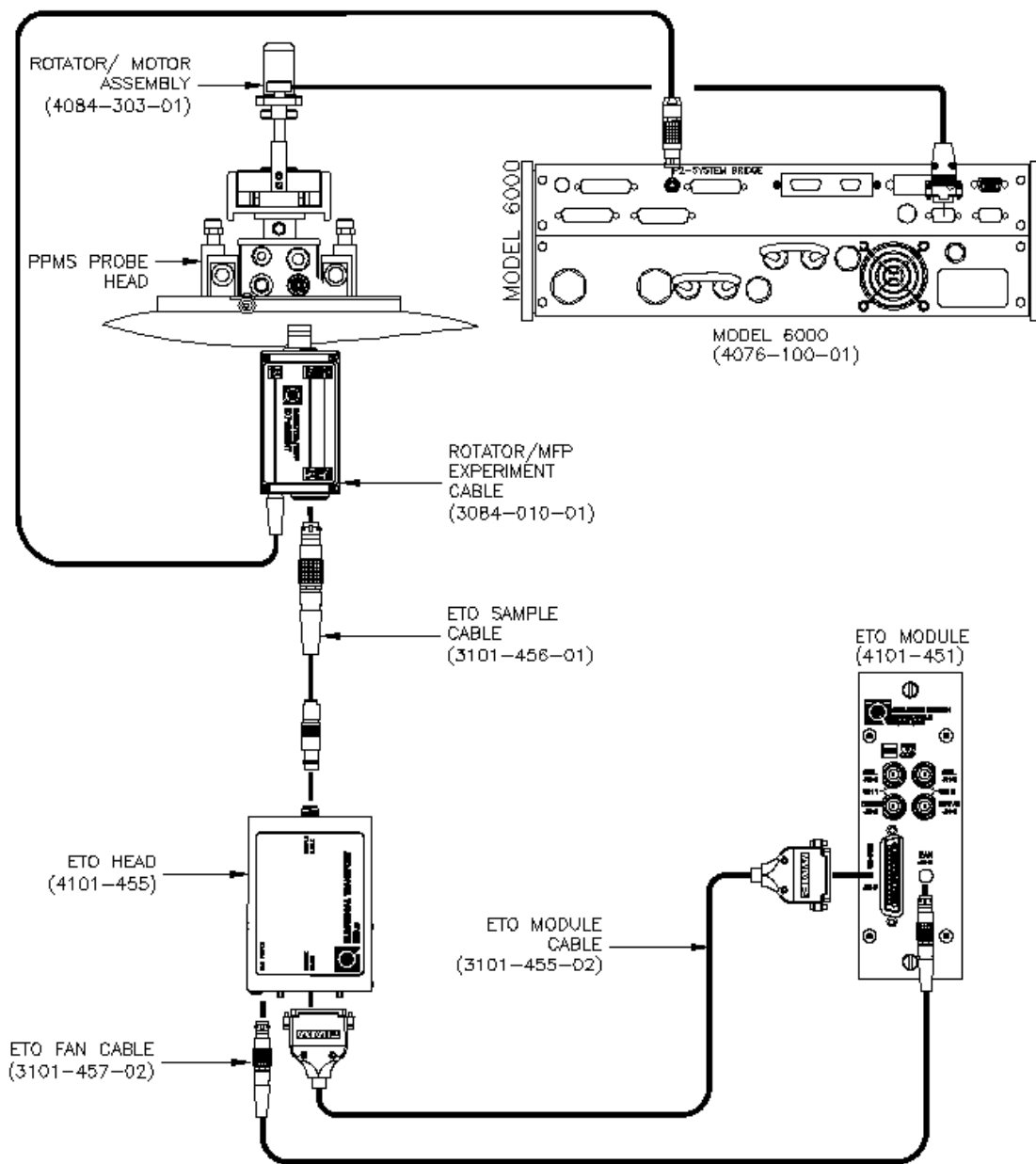


Figure 6-2. ETO and Horizontal Rotator Option Connection Diagram (PPMS)

6.3.2 In PPMS DynaCool or VersaLab with BRT Module

To use the Horizontal Rotator in PPMS Dynacool, you need a BRT module (part number 4101-225) to control the motor module and to read the rotator thermometer and bridge resistances. The BRT module's SROM contains a rotator thermometer table, Low and High resolution motor information, and the rotator serial number. The rotator thermometer can be used if it is plugged into 4 pin Lemo socket connector labeled JL-3 in the BRT module. The Rotator/MFP experiment cable shipped with the Horizontal Rotator allows the rotator thermometer signals to be directed to the bridge board while sample signals are directed to the ETO option.

Complete the following steps to configure the PPMS Dynacool or VersaLab and use the rotator thermometer:

1. Vent the PPMS Dynacool sample chamber and install the rotator/motor assembly (part number 4084-303-05 for Dynacool or -03 for Versalab). Then insert the rotator probe (part number 4084-304 for Dynacool or 4084-305 for Versalab) into the sample chamber.
2. Install the Rotator/MFP experiment cable by plugging the connectors on the cable into the ports indicated by the cable labels. Refer to figure 6-1.
 - Plug the 14-pin gray Lemo connector on the Rotator/MFP experiment cable into the gray, color-coded port on the probe head.
 - Plug the 4-pin Lemo connector on the Rotator/MFP experiment cable labeled "BRT CAN JL-3" into the small round port on the front panel of the BRT module.
 - Plug the 14-pin gray Lemo connector on the ETO sample cable (part number 3101-456-01) into the 14-pin gray Lemo socket on the Rotator/MFP experiment cable box.
3. Note that the Horizontal Rotator is an option in Dynacool, which needs to be activated through the option dialog. Go to "Utilities" and hit "Activate Options..." After activating Rotator, a probe profile is being downloaded from the SROM. Motion control is effective after this activation. In "Rotator Setup" window, select the rotator serial number, motor resolution (Lo-Res or Hi-Res), and enable User Temp. After pressing OK, the temperature control is automatically switched to the User Temp from the Block Temp. In case that the rotator is disconnected unintentionally from the sample tube or a circuit becomes open, the User Temp will switch back to the Block Temp. When the rotator is reconnected, the User Temp will recover from the Block Temp. Data files written by the ETO software now report the rotator thermometer reading, in K.
4. After the measurements are done, deactivate the Rotator option and the Block Temp will become a controlling thermometer.

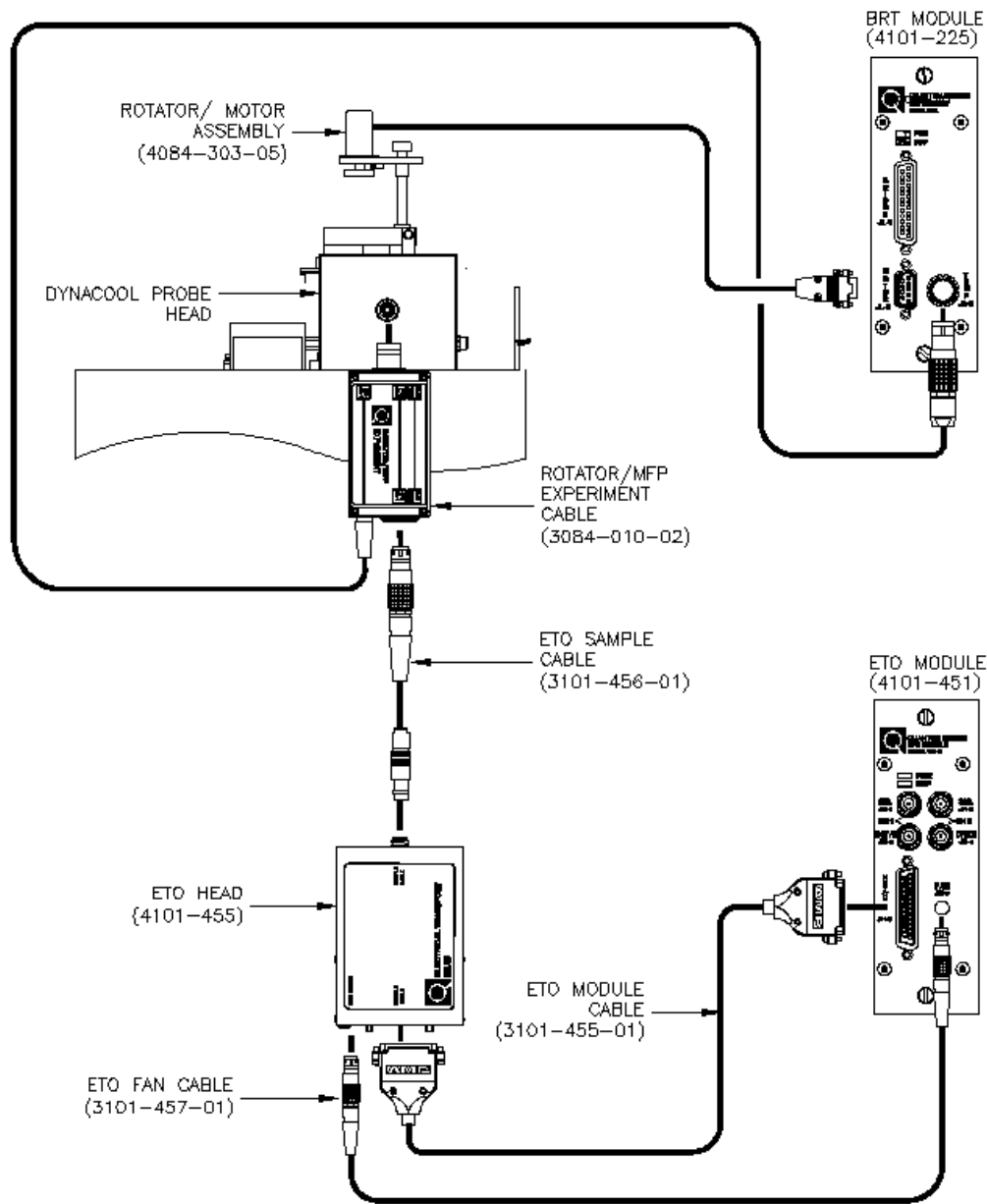


Figure 6-3. ETO and Horizontal Rotator Option Connection Diagram (DynaCool)

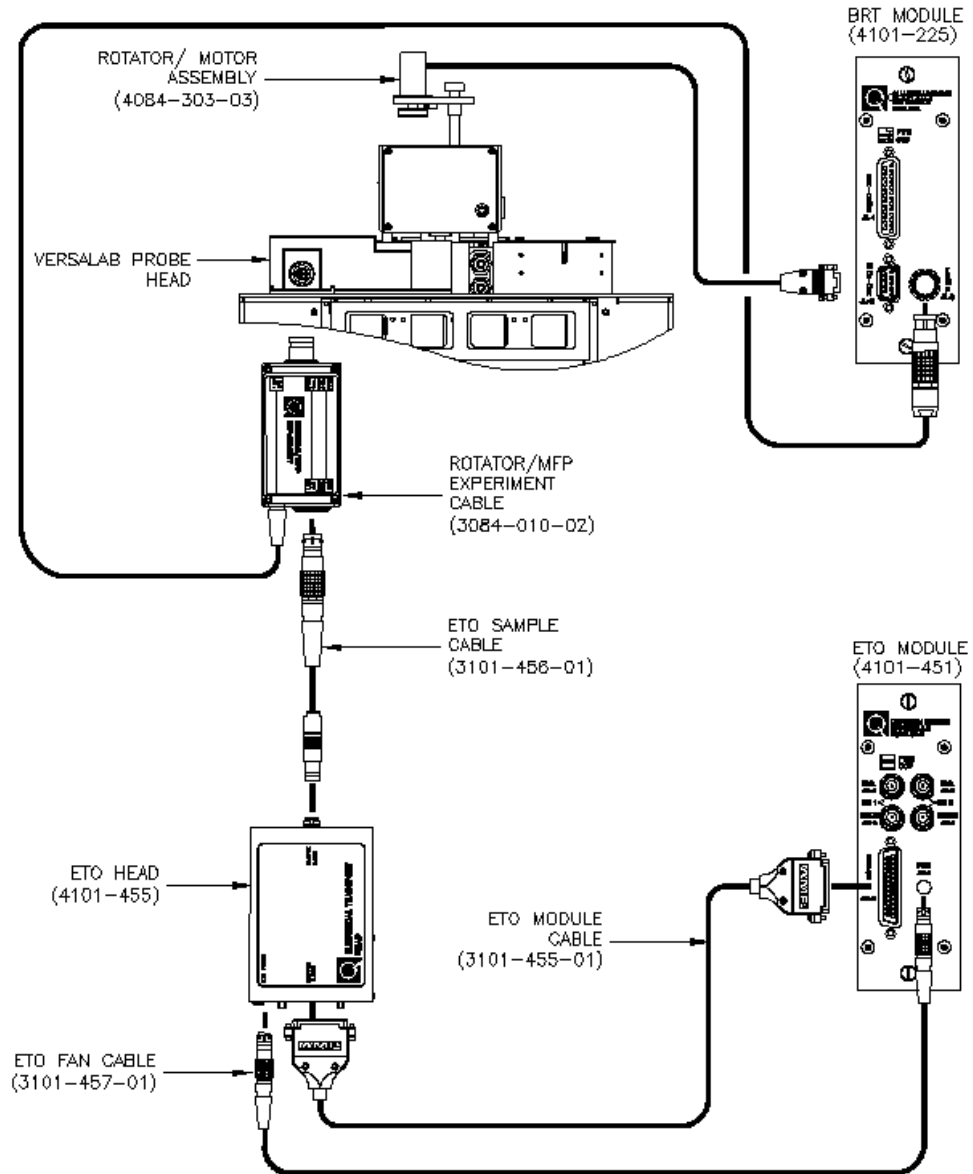


Figure 6-4. ETO and Horizontal Rotator Option Connection Diagram (VersaLab)

6.4 ETO Measurements with the Horizontal Rotator

When the Horizontal Rotator probe is installed in the sample chamber, the ETO option enables users to make several different types of transport measurements over a wide range of resistance values and sample types. Refer to the chapter 1.3 *Electrical Transport Option Measurement Types* for more information about various measurement types. The wiring schemes in figure 7-2 report a positive Hall coefficient if the carriers are hole-like and a negative coefficient if the carriers are electron-like, for samples that are face up in the sample chamber. For Horizontal

Rotators with serial numbers 001–010, the sample is face up at a 180° orientation. Rotators with serial numbers 011 or greater are face upwards with an orientation of 0° or 360°.

Remember to turn off the UserTemp when you remove the Horizontal Rotator probe from the sample chamber. Refer to section 6.3.2

6.5 Interconnection Tables for the ETO/Horizontal Rotator Experiment Cable

Table 6-1. Pin Mapping for P2–System Bridge Port on Model 6000

Sample Holder Board	Gray Lemo Connector On Probe Head	Four-Pin Lemo Connector at P2 Port On Model 6000	Function
3	3	1	Bridge Ch4 I+ (Rotator Therm.)
4	4	2	Bridge Ch4 I– (Rotator Therm.)
5	5	3	Bridge Ch4 V+ (Rotator Therm.)
6	6	4	Bridge Ch4 V– (Rotator Therm.)

Table 6-2. Pin Mapping for sample on ETO/Rotator Experiment Cable

Sample Holder Board	Gray Lemo Connector On Probe Head	Gray Lemo Socket Connector on Rotator Cable	Function
7	7	3	ETO Ch1 I+
8	8	4	ETO Ch1 I-
9	9	5	ETO Ch1 V+
10	10	6	ETO Ch1 V-
11	11	7	ETO Ch2 I+
12	12	8	ETO Ch2 I-
13	13	9	ETO Ch2 V+
14	14	10	ETO Ch2 V-

* Denoted leads on the Horizontal Rotator are a copper alloy, while the remainder of the leads on the Rotator are phosphor-bronze.

Table 6-3. Pin Mapping for JL-3 Bridge Port on BRT Module

Sample Holder Board	Gray Lemo Connector On Probe Head	Four-Pin Lemo Connector at JL-3 on BRT Module	Function
3	3	1	Bridge Ch4 I+ (Rotator Therm.)
4	4	2	Bridge Ch4 I- (Rotator Therm.)
5	5	3	Bridge Ch4 V+ (Rotator Therm.)
6	6	4	Bridge Ch4 V- (Rotator Therm.)

Operation with the Helium-3 System

7.1 Introduction

This chapter contains the following information:

- Section 7.2 presents an overview of Electrical Transport Option Operation with the Helium-3 system.
- Section 7.3 describes how to take ETO Measurements with the Helium-3 System.

7.2 Overview of Electrical Transport Option Operation with the Helium-3 System

The Electrical Transport Option may be used with the PPMS/Dynacool PPMS Helium-3 Refrigerator System insert (Model P825). Use of the Helium-3 insert allows resistance measurements to be performed to below 0.400 K. Although samples are mounted differently when the Helium-3 system is being used, the operation of the Helium-3 system is otherwise designed to be as transparent as possible.

7.2.1 In PPMS with Model 6000

In the GPIB version of Helim-3, the Helium-3 system thermometer is read by channel 4 of the user bridge board using Helium-3 experiment cable (3092-359-01/02). The Helium-3 option uses bridge board channel 3 for reading the Helium-3 system thermometer. Because the thermometer excitation power must be kept very low below 1 K, the enhanced bridge board (part number 3076-050) must be used. As of July 1999, the enhanced bridge board ships standard with all Helium-3 options. The new enhanced bridge board has been designed with a precision-balanced current source to aid precise readings of large resistances.

To set up the system for ETO measurements, the Helium-3 experiment cable is connected in the usual way; that is, between the gray-ringed Lemo connector on the PPMS probe head and the “P1–User Bridge” port on the Model 6000 PPMS Controller. And, the ETO sample cable (3101-456-01/02) is connected to the 14 pin gray Lemo socket connector on the Helium-3 experiment cable box.

7.2.2 In DynaCool with Helium-3 Control Module

In the CAN Version of He3, the thermometer channel is read by a dedicated resistance bridge in Helium-3 module (4101-553) using Helium-3 experiment cable (3092-359-01/02) through a four pin Lemo-type connector on the module's front panel. The Electrical transport measurement is performed with the Electrical transport option with the ETO sample cable (3101-456-02). For more information about the signal wiring any of these options use with the Helium-3 system, please refer to the appropriate option manual.

7.3 ETO Measurements with the Helium-3 System

Please refer to the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* for complete instructions on handling and operating the PPMS Helium-3 system.

Switch to the **Data File** tab to select a new data file for your measurements. Press **Browse** to enter a new file name and location. Checking the *Create New File Version* checkbox will add a numeric suffix to the end of the file name if the file already exists. Press the **View** button to open the data file in MultiVu. **Note:** The data file is not created until a measurement is initiated.

7.3.1 Measurement Setup

7.3.1.1 PREPARE FOR THE MEASUREMENT



WARNING!

Avoid stressing the shaft of the Helium-3 refrigerator probe in any way while you are inserting the refrigerator probe into or removing it from the PPMS sample chamber. The material out of which the probe shaft is constructed is thin and fragile and can easily bend or dent.

1. Remove the Helium-3 refrigerator probe from the sample chamber. Refer to the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* for detailed instructions.
2. Quit the Helium-3 software application if the application is running.

3. Install the ETO sample cable (3101-456-02) from the ETO head (4101-455) by plugging the connectors on the cable into the ports indicated by the cable labels.
 - Plug the 14-pin gray Lemo connector into the gray color socket on the Helium-3 experiment cable box.

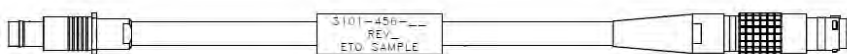


Figure 7-1. ETO Sample Cable

7.3.1.2 INSTALL THE SAMPLE

1. Mount the sample on a Helium-3 sample mount (part number 4092-610).

Note: That at temperatures below 1 K, intimate thermal contact between the sample and the copper holder is very important to reduce temperature errors. A thin film of Apiezon N Grease works well to aid thermal contact. Apiezon N Grease is supplied with the Model P825 Helium-3 insert.

2. Refer to figure 7-2 below to wire the sample to the stage. The plug-in sample mount is clearly labeled for mounting two samples.

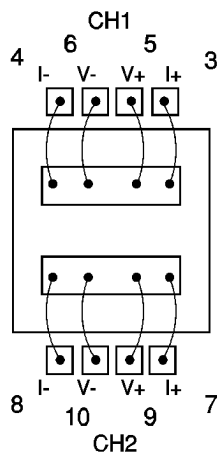


Figure 7-2. Helium-3 sample puck with two samples mounted for four-wire resistance measurements.

3. Attach the plug-in sample stage to the Helium-3 refrigerator probe. Refer to the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* for detailed instructions.
4. Insert the Helium-3 refrigerator probe into the sample chamber. Refer to the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* for detailed instructions.

7.3.1.3 START UP THE SOFTWARE

1. Start-up PPMS MultiVu if the application is not running.

2. Activate the Helium-3 option in PPMS MultiVu. Do the following: (a) select **Utilities > Activate Option**, (b) click on **Helium3** under the **Available Options** heading, and then (c) select the **Activate** button.
3. Activate the Electrical Transport option in PPMS MultiVu. Do the following: (a) select **Utilities > Activate Option**, (b) click on **Resistivity** under the **Available Options** heading, and then (c) select the **Activate** button. Both the Helium-3 software and Electrical Transport option software must be running before you can initiate a measurement.
4. Initiate a Helium-3 system test by using PPMS MultiVu to set an initial temperature (for example, 300 K). The Helium-3 temperature control is activated on the first Temperature Set command. The software checks out the Helium-3 system thermometer and begins controlling temperature.

7.3.2 Performing Measurements

Once the Helium-3 refrigerator probe is inserted in the sample chamber and the Helium-3 and Resistivity software applications are running, you may take resistance measurements at temperatures as low as 0.4 K. You take measurements and write sequences according to the usual procedures described in chapter 4.

7.3.2.1 EXCITATION LEVELS

The sample mounts provided for mounting samples are constructed of high-conductivity copper to provide good thermal anchoring. However, at temperatures below about 1 K, it is normally very difficult to achieve good thermal contact, even within the sample itself. That is, if the excitation current is too high, significant self-heating of the sample can result in substantial temperature errors where the relevant portions of the sample are warmer than the Helium-3 thermometer reports. Again, this is not necessarily due to poor contact of the sample to the sample holder; it may be due to poor thermal conductance of the sample itself. The best way to detect this effect is to repeat measurements using different excitation levels. It is also generally recommended that you use the smallest possible excitation that still gives an adequate signal-to-noise ratio.

Interconnection Tables

A.1 Introduction

This appendix contains the following information:

- Section A.2 shows the standard interconnections for ETO.
- Section A.3 shows the interconnections for ETO/Horizontal Rotator cable.
- Section A.4 shows the interconnections for ETO/Helium-3 User Experiment cable.
- Section A.5 shows the interconnections for ETO/Dilution Refrigerator Experiment cable.

A.2 Standard Interconnection Tables for ETO Sample Cable

Table A-1. Pin Mapping for ETO Sample Cable (3101-456-01/02)

RESISTIVITY SAMPLE PUCK	GRAY LEMO CONNECTOR ON PROBE HEAD	GRAY LEMO CONNECTOR ON ETO SAMPLE CABLE	FUNCTION
3	3	3	ETO Ch1 I+
4	4	4	ETO Ch1 I-
5	5	5	ETO Ch1 V+
6	6	6	ETO Ch1 V-
7	7	7	ETO Ch2 I+
8	8	8	ETO Ch2 I-
9	9	9	ETO Ch2 V+
10	10	10	ETO Ch2 V-
11	11	N/C	N/C
12	12	N/C	N/C
13	13	N/C	N/C
14	14	N/C	N/C

A.3 Interconnection Tables for the ETO/Horizontal Rotator Experiment Cable

Table A-2. Pin Mapping for P2-System Bridge Port on Model 6000

SAMPLE HOLDER BOARD	GRAY LEMO CONNECTOR ON PROBE HEAD	FOUR-PIN LEMO CONNECTOR AT P2 PORT ON MODEL 6000	FUNCTION
3	3	1	Bridge Ch4 I+ (Rotator Therm.)
4	4	2	Bridge Ch4 I- (Rotator Therm.)
5	5	3	Bridge Ch4 V+ (Rotator Therm.)
6	6	4	Bridge Ch4 V- (Rotator Therm.)

Table A-3. Pin Mapping for sample on ETO/Rotator Cable (3084-010-01/02)

SAMPLE HOLDER BOARD	GRAY LEMO CONNECTOR ON PROBE HEAD	GRAY LEMO SOCKET CONNECTOR ON ROTATOR CABLE	FUNCTION
7	7	3	ETO Ch1 I+
8	8	4	ETO Ch1 I-
9	9	5	ETO Ch1 V+
10	10	6	ETO Ch1 V-
11	11	7	ETO Ch2 I+
12	12	8	ETO Ch2 I-
13	13	9	ETO Ch2 V+
14	14	10	ETO Ch2 V-

* Denoted leads on the Horizontal Rotator are a copper alloy, while the remainder of the leads on the Rotator are phosphor-bronze.

A.4 Interconnection Tables for the ETO/Helium-3 User Experiment Cable

Table A-4. Pin Mapping for sample on ETO/Helium-3 user Cable (3092-359-01/02)

HELIUM-3 PROBE CONNECTOR	HELIUM-3 SAMPLE MOUNT	GRAY LEMO CONNECTOR ON PROBE HEAD	GRAY LEMO SOCKET CONNECTOR ON HELIUM-3 USER CABLE	FUNCTION
3	3	3	3	ETO Ch1 I+ (User/resist)
4	4	4	4	ETO Ch1 I- (User/resist)
5	5	5	5	ETO Ch1 V+ (User/resist)
6	6	6	6	ETO Ch1 V- (User/resist)
7	7	7	7	ETO Ch2 I+ (User/resist)
8	8	8	8	ETO Ch2 I- (User/resist)
9	9	9	9	ETO Ch2 V+ (User/resist)
10	10	10	10	ETO Ch2 V-

* Denoted leads on the Horizontal Rotator are a copper alloy, while the remainder of the leads on the Rotator are phosphor-bronze.

Table A-5. Pin Mapping of Helium-3 Thermometer on Model 6000 for PPMS

HELIUM-3 PROBE CONNECTOR	GRAY LEMO CONNECTOR ON PROBE HEAD	FOUR-PIN LEMO CONNECTOR AT P1 PORT ON MODEL 6000	FUNCTION
11	11	1	Bridge Ch4 I+ (Helim-3 Therm.)
12	12	2	Bridge Ch4 I- (Helim-3 Therm.)
13	13	3	Bridge Ch4 V+ (Helim-3 Therm.)
14	14	4	Bridge Ch4 V- (Helim-3 Therm.)

Table A-6. Pin Mapping of Helium-3 Thermometer on HE3 Control Module for PPMS Dynacool

HELIUM-3 PROBE CONNECTOR	GRAY LEMO CONNECTOR ON PROBE HEAD	FOUR-PIN LEMO SOCKET CONNECTOR ON HELIUM-3 USER CABLE	FOUR-PIN LEMO CONNECTOR AT HELIUM-3 CONTROL MODULE	FUNCTION
11	11	1	1	Bridge Ch4 I+ (Helim-3 Therm.)
12	12	2	2	Bridge Ch4 I- (Helim-3 Therm.)
13	13	3	3	Bridge Ch4 V+ (Helim-3 Therm.)
14	14	4	4	Bridge Ch4 V- (Helim-3 Therm.)

A.5 Interconnection Tables for the ETO/Dilution Refrigerator (DR) Experiment Cable

Table A-7. Pin Mapping for ETO sample, Dilution Refrigerator Cable (3092-626)

10-PIN FISCHER AT PROBE HEAD LABELED SAMPLE	10-PIN FISCHER SOCKET AT ETO SAMPLE, DR CABLE	DR SAMPLE STAGE CONNECTION	FUNCTION
1	1	N/C	
2	2	N/C	
3	3	3	ETO Ch1 I+ (User/resist)
4	4	4	ETO Ch1 I- (User/resist)
5	5	5	ETO Ch1 V+ (User/resist)
6	6	6	ETO Ch1 V- (User/resist)
7	7	7	ETO Ch2 I+ (User/resist)

Table Continued...

10-PIN FISCHER AT PROBE HEAD LABELED SAMPLE	10-PIN FISCHER SOCKET AT ETO SAMPLE, DR CABLE	DR SAMPLE STAGE CONNECTION	FUNCTION
8	8	8	ETO Ch2 I- (User/resist)
9	9	9	ETO Ch2 V+ (User/resist)
10	10	10	ETO Ch2 V-

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



Physical Property Measurement System

Thermal Transport Option User's Manual

Part Number 1684-100B

Quantum Design

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Third edition of manual completed October 2002.

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U.S. Patents

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)
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

C O N T E N T S

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.

P.2 Scope of the Manual

This manual discusses the Thermal Transport option (TTO) for the Physical Property Measurement System (PPMS). This manual explains how to use the TTO system and it explains the theory of operation for TTO. This manual describes the hardware and software that are unique to TTO, and it includes maintenance and troubleshooting information.


For detailed information about the PPMS MultiVu software, which is the parent software application running the PPMS, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual*.

P.3 Contents of the Manual

- Chapter 1 presents an overview of the TTO system and of the TTO theory of operation.
- Chapter 2 discusses and illustrates the hardware used with TTO.
- Chapter 3 discusses the TTO software and TTO data files.
- Chapter 4 explains how to prepare samples for TTO measurements.
- Chapter 5 explains how to take measurements with TTO and describes the measurement process.
- Chapter 6 contains troubleshooting suggestions.
- Chapter 7 explains basic maintenance procedures.

- Appendix A explains how to install the TTO hardware and software.
- Appendix B contains status codes and error messages.
- Appendix C contains pinout tables.

P.4 Conventions in the Manual

File menu	Bold text distinguishes the names of menus, options, buttons, and panels appearing on the PC monitor or on the Model 6000 PPMS Controller LCD screen.
File>Open	The > symbol indicates that you select multiple, nested software options.
STATUS	Bold text and all CAPITAL letters distinguish the names of keys located on the front panel of the Model 6000 PPMS Controller.
<code>.dat</code>	The <i>Courier</i> font distinguishes characters you enter from the PC keyboard or from the Model 6000 PPMS Controller front panel. The <i>Courier</i> font also distinguishes code and the names of files and directories.
<Enter>	Angle brackets <> distinguish the names of keys located on the PC keyboard.
<Alt+Enter>	A plus sign + connecting the names of two or more keys distinguishes keys you press simultaneously.
 NOTE	A pointing hand and the word NOTE introduce a supplementary note.
CAUTION!	Cautionary notes are preceded with the word CAUTION! This signals conditions that could result in loss of information or damage to your equipment.
WARNING!	Warnings are preceded with the word WARNING! This signals conditions that could result in bodily harm or loss of life.

Introduction

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the TTO system.
- Section 1.3 describes the TTO system measurement modes.
- Section 1.4 explains how the TTO system measures thermal properties.
- Section 1.5 explains the TTO system's theory of operation.
- Section 1.6 contains the start-up checklist for secondary installation of the TTO.

1.2 Overview of the Thermal Transport Option

The Quantum Design Thermal Transport option (TTO) for the Physical Property Measurement System (PPMS) enables measurements of thermal properties, including thermal conductivity κ and Seebeck coefficient (also called the thermopower) α , for sample materials over the entire temperature and magnetic field range of the PPMS. The TTO system measures thermal conductivity, or the ability of a material to conduct heat, by monitoring the temperature drop along the sample as a known amount of heat passes through the sample. TTO measures the thermoelectric Seebeck effect as an electrical voltage drop that accompanies a temperature drop across certain materials. The TTO system can perform these two measurements simultaneously by monitoring both the temperature and voltage drop across a sample as a heat pulse is applied to one end. The system can also measure electrical resistivity ρ by using the standard four-probe resistivity provided by the PPMS AC Transport Measurement System (ACT) option (Model P600). All three measurement types are essential in order to assess the so-called “thermoelectric figure of merit,” $ZT = \alpha^2 T / \kappa \rho$, which is the quantity of main interest if you are investigating thermoelectric materials.

While the measurements taken with the TTO system are quite elementary in principle, they have eluded commercialization because the data was typically very error prone, time consuming, and laborious, due—for example—to problems in controlling heat flow and accurately measuring small temperature differentials in a convenient manner. The TTO system has solved or greatly reduced many of these experimental complications. TTO uses convenient sample mounting, small and highly accurate Cernox chip thermometers, and sophisticated software that dynamically models an AC heat flow through the sample and corrects for any heat losses that occur. The PPMS with the High-Vacuum option (Model P640) provides an ideal environment for the custom-designed TTO sample puck, and the ACT option (Model P600) powers the sample heater and takes resistivity measurements. Table 1-1 on the following page lists the TTO system requirements.

Table 1-1. System Requirements for the Thermal Transport System*

COMPONENT	FUNCTION
PPMS Resistivity Option (Model P400)	Provides user bridge board that reads two thermometer shoes.
PPMS AC Transport Measurement System (Model P600)	Outputs current to heater and sample while providing low-noise, phase-sensitive detection.
PPMS High-Vacuum Option (Model P640)	Provides thermal isolation for measurements. Cryopump or Turbo Pump may be used.
PPMS MultiVu Software Version 1.1.6 or Later	Provides single user interface for PPMS and PPMS options.

* In addition to the requirements in Table 1-1, the PPMS Continuous Low-Temperature Control (CLTC) option (Model P800) is highly recommended. CLTC provides extended low-temperature control.

Table 1-2. Thermal Transport System Parameters

PARAMETER	VALUE
Pressure	High vacuum ($\sim 10^{-4}$ torr)
Temperature	1.9–390 K
Magnetic field	0–14 T when $T > 20$ K

If you require use of significant magnetic fields ($H > 0.1$ T) at temperatures below $T \sim 20$ K, please inquire with Quantum Design.

Table 1-3. Thermal Transport System Components

COMPONENT	PART NUMBER	ILLUSTRATION
Thermal Transport sample puck including Isothermal radiation shield	4084-570 4084-575 4084-579	Figure 2-1
Two plug-in thermometer shoes	4084-580T	Figure 2-1
Plug-in heater shoe	4084-585	Figure 2-1
User's kit	4084-569	Figure 2-3
Two nickel standard samples	4084-593	Figure 2-3
WaveROM EPROM for AC board	3084-043	Figure 2-4
Thermal Transport connection cable	3084-582	Figure 2-5
Thermal Transport software module		

1.2.1 Purpose of Measuring Thermal Transport Properties

In measuring the thermal transport properties of a material specimen—such as thermal conductivity κ and Seebeck coefficient α —a researcher can learn considerable information about the electronic as well as the ionic lattice structure of that specimen. Thermal conductivity is a measure of the ability of a material to conduct heat, so measuring this quantity provides information about scattering of heat-carrying phonons and electrons. The Seebeck coefficient describes the thermal diffusion of free charge carriers (electrons or holes), which creates an electric field inside a material when a temperature gradient is sustained. Much like the electrical resistivity, this property is very sensitive to subtle changes in the electronic scattering processes and can be a powerful probe in that regard.

Taken together with electrical resistivity ρ , the thermal conductivity and Seebeck coefficient also provide a measure of the so-called thermoelectric figure of merit $Z = \alpha^2/(\kappa\rho)$, which is a quantity of practical significance because it quantifies a material's ability to transport heat by the application of an electric current (Peltier effect), or conversely, a material's ability to generate an electric field by passing a thermal current (Seebeck effect, described above). The figure of merit is usually expressed as the dimensionless quantity $Z \times T$, where $Z \times T \sim 1$ is a common benchmark for viability of a material for thermoelectric applications.

1.3 Measurement Modes

The TTO system includes two measurement modes:

- Continuous measurement mode
- Single measurement mode

All properties measurements offered by TTO can be performed in either of these two modes. Parameters for each measurement mode (see Section 5.3) may be specified prior to running any measurement in that mode.

1.3.1 Continuous Measurement Mode

In continuous measurement mode, measurements are being taken continually and the adaptive software is adjusting parameters (such as heater power and period) to optimize the measurements. This mode is amenable to slow sweeps of system variables such as temperature or magnetic field, and it is often the most rapid way of obtaining data because you do not have to wait for the system to reach equilibrium before measuring. The continuous mode is also expedited by the use of a sophisticated curve-fitting algorithm that determines the steady-state thermal properties by extrapolating from the response to a relatively short (typically several minutes) heat pulse.

1.3.2 Single Measurement Mode

The single measurement mode is slower than continuous measurement mode because it requires that the system reach a steady state in both the heater “off” and “on” states, which also implies that temperature or field slewing is unavailable. The advantage of single measurement mode is that no

subtle curve-fitting calculations are required, so interpretation of the raw data is in principle more straightforward. Researchers who study thermal transport properties usually employ this steady-state technique because of its simplicity and robustness. In either style of single measurement—stability or timed—described below in Table 1-4, data is first taken in the heater “off” state once the system settles. After the user-specified heater power is applied, the system waits for the selected equilibrium condition before making the final measurement in the heater “on” state. You can view the live ΔT vs. time data in the **Waveform** tab of the Thermal Transport control center to monitor measurement progress.

Table 1-4. Styles for Measurements Taken in Single Measurement Mode

MEASUREMENT STYLE	DEFINITION
Stability	System takes first measurement in heater “off” state once temperature stability at both hot and cold sample thermometers is within a specified window, stated either as a percentage of T or as an absolute number (in kelvin). After heat is applied, system waits for the same stability criterion to be met before taking final measurement. Heater power is turned off after conclusion of this measurement. User-specified timeout forces system to take a measurement at timeout period even if stability criterion has not been met.
Timed	Sends heat pulse of user-specified duration into sample. System takes a measurement of temperatures and thermal voltages before applying heat, and then takes final measurement at end of heat pulse.

1.4 Measured Thermal Properties

The TTO system is set up to measure four thermal transport properties:

- Thermal conductivity
- Seebeck coefficient
- Electrical resistivity
- Thermoelectric figure of merit

If thermal conductivity, Seebeck coefficient, and electrical resistivity are all measured, then the thermoelectric figure of merit, which is the algebraic combination of these three measurements, can be determined.

Separate measurement protocols are provided for thermal conductivity, Seebeck coefficient, and electrical resistivity because these individual quantities may be more accurately measured by using excitation currents and temperature differentials optimized for each situation. Limits for the parameters defining each measurement may be specified prior to running the measurement. Section 5.3 discusses the measurement parameters.

Each measured thermal transport property may be determined in either of the two measurement modes (continuous or single) supported by the TTO system; refer to Section 1.3. You select a measurement mode, and then you select the thermal properties to measure in that mode.

1.4.1 Thermal Conductivity

The TTO system measures thermal conductivity κ by applying heat from the heater shoe in order to create a user-specified temperature differential between the two thermometer shoes. The TTO system dynamically models the thermal response of the sample to the low-frequency, square-wave heat pulse, thus expediting data acquisition. TTO can then calculate thermal conductivity directly from the applied heater power, resulting ΔT , and sample geometry.

1.4.2 Seebeck Coefficient

The TTO system determines the Seebeck coefficient (also called the thermopower) α by creating a specified temperature drop between the two thermometer shoes—just as it does to measure thermal conductivity. However, for Seebeck coefficient the voltage drop created between the thermometer shoes is also monitored. The additional voltage-sense leads on these thermometer shoes are connected to the ultra-low-noise preamplifier of the ACT system.

1.4.3 Electrical Resistivity

The TTO system measures electrical resistivity ρ by using a precision DSP current source and phase-sensitive voltage detection. The specifications for this AC resistivity measurement are essentially identical to those for the AC Transport Measurement System (ACT) option, because the same high-performance hardware is used by both TTO and ACT. The *Physical Property Measurement System: AC Transport Option User's Manual* discusses the ACT measurements in detail.

1.4.4 Figure of Merit

The dimensionless thermoelectric figure of merit ZT is determined here simply as the algebraic combination $ZT = \alpha^2 T / \kappa \rho$ of the three measured quantities—thermal conductivity, Seebeck coefficient, and electrical resistivity—discussed above.

1.5 Theory of Operation

Benefits of the design of the TTO system include the following:

- Four-terminal geometry minimizes the effects of thermal and electrical resistance of the leads
- Continuous measurements while slewing in temperature provide high density of data
- Careful attention to the removal of effects of temperature drift, thermal radiation, and other systematic errors
- Robust, easy-to-use, fully automated measurements

1.5.1 Hardware

When measuring in continuous mode, the DSP hardware in the Model 7100 AC Transport Controller generates the heat pulse in the chip resistor heater on the sample, which can be described as an “on” cycle of constant power followed by an “off” cycle of equal duration. The waveform for this pulse was programmed specially for the TTO system in the waveROM EPROM on the AC board, so older AC boards must have the old waveROM swapped for the new waveROM (labeled “THRMXPT 4201”) to run TTO. Section 2.2.4 discusses the waveROM EPROM in more detail.

Figure 1-2 on the following page illustrates the heat pulse as well as the temperature and voltage response at the hot and cold thermometer shoes in an idealized sample.

1.5.2 Thermal and Electrical Circuit

The thermal and electrical connections for an idealized TTO sample are shown in Figure 1-1. For clarity, the sample is shown mounted in the four-probe geometry. The four basic physical elements are illustrated: the sample, the epoxy bonds that adhere the leads to the sample, the copper leads, and the heater and thermometer shoe assemblies that screw down onto the leads. For thermal conductivity and Seebeck coefficient measurements, heat is applied to one end of the sample by running current through the heater ($Q_{+/-}$). The temperatures T_{hot} and T_{cold} are measured at the thermometer shoes. Also during the heat pulse, the Seebeck voltage ($\Delta V = V_+ - V_-$) is monitored. Heat exits the sample to the coldfoot. Time traces of ΔT and ΔV during the heat pulse are illustrated in Figure 1-2.

Electrical resistivity measurements are made both before and after the heat pulse described above. Current ($I_{+/-}$) flows through the sample and the voltage drop across the sample is monitored using the $V_{+/-}$ leads.

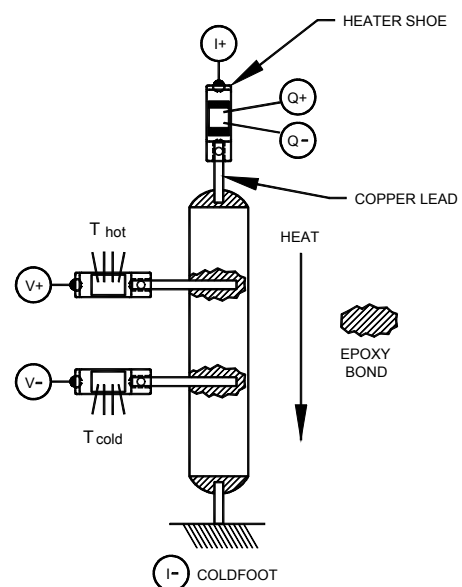


Figure 1-1. Thermal and Electrical Connections for an Idealized Sample

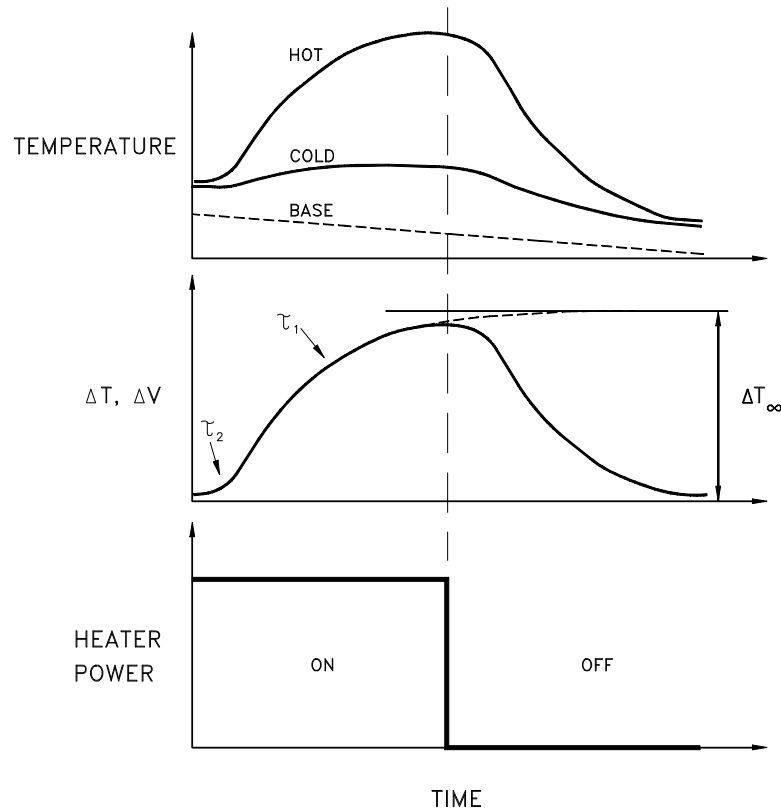


Figure 1-2. Heat Pulse and Temperature and Voltage Response at Hot and Cold Thermometer Shoes in an Idealized Sample

- Top panel: Time trace of hot and cold thermometers during an idealized heat pulse; note that the PPMS base temperature is slewing.
- Middle panel: Corresponding temperature ΔT and voltage ΔV differentials across the sample, indicating thermal time constants τ_1 and τ_2 and the estimate of the asymptotic differential ΔT_∞ .
- Bottom panel: Heater power during square-wave heat pulse.

1.5.3 Software Models

In continuous measurement mode (Section 1.3.1), the software uses adaptive algorithms to optimize measurement parameters such as heater current, heat pulse period, and resistivity excitation amplitude and frequency. Once the ΔT vs. time data over the duration of the heat pulse is obtained, a nonlinear least-squares fitting routine, which fits the data to the empirical formula, is launched:

$$\Delta T_{\text{model}} = \Delta T_\infty \times \{1 - [\tau_1 \times \exp(-t/\tau_1) - \tau_2 \times \exp(-t/\tau_2)]/(\tau_1 - \tau_2)\} \quad (\text{Equation 1-1})$$

where ΔT_∞ represents the asymptotic temperature drop across the sample if the heater is left on indefinitely, and τ_1 and τ_2 are long and short empirical time constants, respectively, for the sample (see Figure 1-2). The fitting routine performs an exhaustive search over the space of these three parameters, reducing the space iteratively until the parameter values that yield the minimum in the residual of the curve fit are identified satisfactorily. Equation 1-1 is appropriate to the data taken during the heating pulse, while the data taken during the cooling pulse is simultaneously fit essentially by changing the sign of the model equation: $\Delta T_{\text{model,cooling}} = A - \Delta T_{\text{model,heating}}$, where A is a constant. Due to long

thermal diffusion times (τ_1), the thermal history of the sample must be accounted for in the model, and this is achieved by including the remanent effects of the two previous pulses in modeling the current pulse.

The fitting routine for Seebeck coefficient data is similar, yet it is less computationally intensive. The ΔV vs. time data is read back from the DSP buffer at the end of the measurement, and after the ΔT vs. time data is fit to obtain τ_1 and τ_2 , a *linear* least-squares routine fits the data to the following equation:

$$\Delta V_{\text{model}} = \Delta V_{\infty} \times \{1 - [\tau_1 \times \exp(-t/\tau_1) \pm \tau_2' \times \exp(-t/\tau_2')]/(\tau_1 - \tau_2')\} + bt + c \quad (\text{Equation 1-2})$$

where ΔV_{∞} is the asymptotic Seebeck voltage drop akin to ΔT_{∞} in equation 1-1, b and c are parameters that describe linear drift and offset voltages, respectively, and $\tau_2' = 0 \dots \tau_1$ is swept so that for each value of τ_2' a linear regression in ΔV_{∞} , b , and c is performed. Note that “ \pm ” is used between the exponential terms and signifies that a full search is done for each sign. The physical significance of this is that the Seebeck coefficient of the material responsible for the short time constant τ_2 (that is, the leads) may be of the opposite sign as that for the material associated with the long time constant (that is, the sample). This is in contrast to the case of the thermal conductivity, which is always positive. The parameter for the linear voltage drift b is included here to account for varying thermal voltages in the wiring to the sample and also the slow microvolt-level drift in the preamp electronics.

A similar measurement technique, previously published by Maldonado¹, describes modeling of the thermal and thermoelectric response of a sample to a low-frequency, square-wave heat pulse. However, the thermal circuit considered in that work was considerably simpler than that which is appropriate to TTO, and hence the modeling was done differently.

1.5.4 Estimating Errors in the Data

The software also estimates the standard deviations (σ) in the reported quantities of thermal conductivity, Seebeck coefficient, electrical resistivity, and figure of merit ZT. This is done by estimating the goodness of the curve fits to κ , α , and ρ by calculating the residual of the curve fit. We make the assumption that this residual reflects the error in our estimate of the quantity (DT or DV), and this is true when the data deviates from the curve fit in a random manner. If deviations are systematic, as can be seen by inspecting the data in the `.raw` file, this indicates that the curve fit does not properly represent the data and the quantity and that error estimates are incorrect. If this occurs, consult Chapter 6 for troubleshooting tips. The residual for the ΔT vs. time curve fitting is calculated as follows:

$$\text{Residual} = R_{\Delta T} = \sqrt{\frac{\sum_i (\Delta T_i - \Delta T_{i,\text{model}})^2}{N}} \quad (\text{Equation 1-3})$$

where N is the number of data points making up the curve. In the measurements of κ and α , $N = 64$, while for ρ , $N = 128$. Since the thermal conductance $K = P/\Delta T$, errors in the heater power P (see the next section) must also be taken into account. The standard deviation in the conductivity is then calculated:

$$\sigma(\kappa) = \kappa \times \sqrt{\left(\frac{R_{\Delta T}}{\Delta T_{\infty}}\right)^2 + \left(\frac{2IR\delta I}{P}\right)^2 + \left(\frac{0.2 \times P_{\text{loss}}}{P}\right)^2 + \left(\frac{0.1 \times T_{\infty} \times K_{\text{shoes}}}{P}\right)^2} \quad (\text{Equation 1-4})$$

¹ Maldonado, O. Pulse method for simultaneous measurement of electric thermopower and heat conductivity at low temperatures. *Cryogenics*, vol. 32, (no. 10), 1992. 908–12.

The first term is the residual of the curve fit mentioned above, the second term propagates the error in the heater current I (heater resistance is R) due to the digital-analog converter, the third term is the error in the estimation of the sample radiation term where 20% combined error in the estimation of sample surface area and emissivity is assumed, and the last term is the error in the thermal conductance leak from the shoe assemblies K_{shoes} where a 10% error in this correction is assumed (see the next section for details on heat losses).

The error in the measurement of the thermal voltage ΔV vs. time has a similar expression as equation 1-3, so the standard deviation in the Seebeck coefficient $\alpha = \Delta V/\Delta T$ is the following:

$$\sigma(\alpha) = \alpha \times \sqrt{\left(\frac{R_{\Delta V}}{\Delta V_{\infty}}\right)^2 + \left(\frac{R_{\Delta T}}{\Delta T_{\infty}}\right)^2} \quad (\text{Equation 1-5})$$

Resistivity measurements are made both preceding and following each thermal measurement so that the average of the two ρ and $\sigma(\rho)$ values is reported in the data file. The residual of the curve fits R_p is obtained from the stream of voltage V vs. time data as the following:

$$\text{Residual} = R_p = \sqrt{\frac{\sum_i (V_i - V_{i,\text{model}})^2}{N}} \quad (\text{Equation 1-6})$$

and the standard deviation is calculated simply as

$$\sigma(\rho) = \rho \times \frac{R_p}{V_{PP}} \quad (\text{Equation 1-7})$$

where V_{PP} is the peak-to-peak amplitude of the voltage vs. time signal.

The standard deviation in the figure of merit ZT is obtained by propagating the errors from each of the measurements:

$$\sigma(ZT) = ZT \times \sqrt{\left(\frac{2\sigma(\alpha)}{\alpha}\right)^2 + \left(\frac{\sigma(\kappa)}{\kappa}\right)^2 + \left(\frac{\sigma(\rho)}{\rho}\right)^2 + \left(\frac{\sigma(T)}{T}\right)^2} \quad (\text{Equation 1-8})$$

where the last term is the standard deviation of the sample temperature over the measurement.

1.5.5 Correcting for Heat Loss

Thermal conductance is determined as $K = P/\Delta T$ where P is the heat flowing through the sample. Since the heat flux cannot be measured directly, the net conducted heat through the sample is estimated as the power (I^2R) dissipated in the heater resistor, minus losses due to radiation or thermal conduction down the leads from the shoe assemblies. Thus the conductance is determined as follows:

$$K \text{ [W/K]} = (I^2R - P_{\text{rad}}) / \Delta T - K_{\text{shoes}} \quad (\text{Equation 1-9})$$

where

$$K_{\text{shoes}} = aT + bT^2 + cT^3 \quad (\text{Equation 1-10})$$

is a standard estimate of the thermal conductance of the shoe assemblies (a , b , and c are constants), and

$$P_{\text{rad}} = \sigma_T \times (S/2) \times \varepsilon \times (T_{\text{hot}}^4 - T_{\text{cold}}^4) \quad (\text{Equation 1-11})$$

is the radiation from the sample, S is the total sample surface area, ε is the infrared emissivity of the radiating surface (see Section 5.2.2 for more information on estimating the emissivity), $T_{\text{hot/cold}}$ are the average temperatures of the hot and cold thermometers during the measurement, and $\sigma_T = 5.67 \times 10^{-8} \text{ W m}^{-2} \text{ K}^{-4}$ is the Stefan-Boltzmann constant. The factor of $1/2$ in the equation is due to the approximation that only half of the sample surface area is radiating at the hot temperature, while the other half is at the cold temperature.

Since radiative heat losses are often very difficult to accurately estimate, you should expect errors in measurements of thermal conductance above $T \sim 300 \text{ K}$ that are on the order of $\pm 1 \text{ mW/K}$.

1.5.6 Correcting for Seebeck Coefficient of Manganin Leads

The manganin leads that connect the shoes to the connector plugs have a small Seebeck coefficient (no more than $\sim 1 \text{ } \mu\text{V/K}$ at any temperature), and this has been estimated and subtracted from the “Seebeck Coef. ($\mu\text{V/K}$)” data column in the data file. However, the column “Seebeck Volt. (μV)” is uncorrected.

1.6 Start-up Checklist for Secondary Installation

1. Verify that the new AC board with the waveROM EPROM is installed in the Model 6000 PPMS Controller. Refer to Sections 2.2.4 and 2.2.4.1.
2. Verify that the Resistivity option (Model P400) is installed and the user bridge board is in the Model 6000 PPMS Controller.
3. Verify that all proper connections are made between the Model 6000 PPMS Controller and the Model 7100 AC Transport Controller. Refer to Figure A-1.
4. Verify that the High-Vacuum option (Model P640) is installed and activated.
5. Verify that the gray Lemo cable for the Thermal Transport option is properly connected.
6. Verify that the TTO software is installed as a PPMS MultiVu option.

Hardware

2.1 Introduction

This chapter contains the following information:

- Section 2.2 discusses and illustrates the TTO hardware.
- Section 2.3 discusses the ACT option hardware that is used with TTO.
- Section 2.4 discusses the High-Vacuum option hardware that is used with TTO.
- Section 2.5 explains how to calibrate new shoe assemblies.

2.2 Thermal Transport Hardware

The TTO hardware includes the following:

- Thermal Transport sample puck
- Isothermal radiation shield (tube and cap)
- Two calibrated plug-in thermometer shoes
- One calibrated plug-in heater shoe
- Three uncalibrated shoes: two thermometer shoes and one heater shoe
- User's kit
- Two nickel calibration samples
- Thermal Transport connection cable

The TTO system also uses ACT hardware (Section 2.3), High-Vacuum hardware (Section 2.4), and, if required, an AC board ROM upgrade kit.

2.2.1 Thermal Transport Sample Puck

The Thermal Transport sample puck (Figure 2-1) plugs into the 12-pin socket at the bottom of the PPMS sample chamber. The Thermal Transport puck is inserted into the sample chamber by using the standard PPMS puck extraction tool (part number 4084-110). All 12 pins on the puck are used for thermal transport measurements (Appendix C lists pinouts). The puck serial number is written on the plastic socket of the base.

Modularized shoe assemblies, including two temperature/voltage shoes and one heater/current shoe, on the Thermal Transport puck connect to the three five-pin sockets on the green printed circuit board. Each gold-plated copper “shoe” has a hole in which the appropriate sample lead is inserted and held in the shoe by a small stainless steel metric M1 screw. The temperature/voltage shoe assemblies contain a Cernox 1050 thermometer as well as a voltage lead that is soldered to the shoe itself. The heater/current shoe assembly contains a resistive heater chip as well as an electrical current source lead (I+) that is soldered to the shoe. At the other end of each shoe assembly is a five-pin electrical plug on which the serial number is written. Each shoe type—heater or thermometer—is individually serialized. The 2-inch-long, 0.003-inch-diameter wires used for leads on the shoe assemblies are designed to minimize thermal conduction from the sample to the puck, and hence all are made of manganin alloy with the exception of the current (I+) lead, which is made of PD-135 low-resistance copper alloy. Two Sharpie permanent markers, red and blue, have been included with the TTO system so that you can color the alumina Cernox chip housing on each thermometer shoe, as well as the plastic electrical plug at the other end, to indicate the hot and cold probes. Marking the housing is convenient because it is easy to confuse the two sets of wires between the sockets and the shoes. The sides of the sockets for the thermometer shoe assemblies are painted so that the middle socket is red (hot probe) and the left-side socket is blue (cold probe). The right-hand socket, which is unpainted, is used only by the heater shoe assembly.

The sample is connected to the puck at the coldfoot, which contains a Phillips screw and a stainless steel clamp on the bottom that clamps onto the sample lead. This is the thermal sink for the sample, so good thermal contact is important here. If achieving good thermal contact is a concern, a small amount of Apiezon H Grease, which is included in the TTO user’s kit (Section 2.2.2), can be used on the sample lead to improve contact to the coldfoot. Note that good electrical contact is also required at the coldfoot if resistivity measurements are being made.

The copper isothermal radiation shield screws into the base of the puck and is designed to minimize radiation between the sample and the environment. The cap is removable so that you can verify that the leads and the sample do not touch the shield. A copper shield plate is also placed between the sample stage and the PC board sockets to minimize radiation effects.

CAUTION! Use care when threading the radiation shield onto the puck. The copper metal is soft, so excessive force or misthreading of the piece can easily damage the threads.

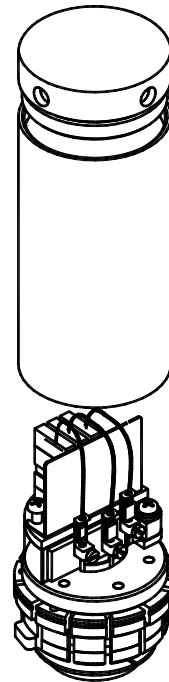


Figure 2-1. TTO Puck with Radiation Shield

2.2.2 User's Kit

The user's kit contains miscellaneous hardware and consumables that are needed for mounting leads on samples as well as calibrating the spare shoe assemblies provided with the Thermal Transport option. The convenient portable toolbox (see Figure 2-3 on the following page) helps keep the items in the kit organized.

The contents of the user's kit include the following.

- **Puck-mounting station**
A pivoting, rotating socket is mounted to a heavy base (Figure 2-2) and holds the Thermal Transport sample puck in a fixed position, giving you better access to the sample leads while you are connecting the shoes or making other adjustments. Tighten the two thumbscrews on the mounting station once the desired orientation for the puck is achieved.

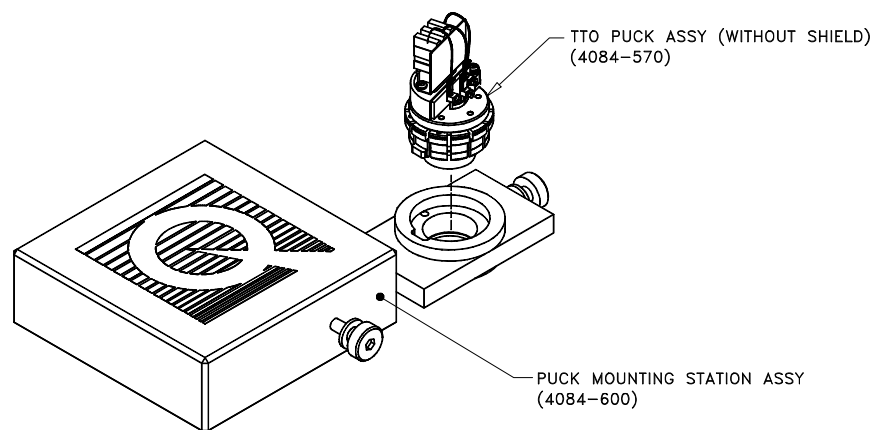


Figure 2-2. Puck-Mounting Station with Puck

- **Nickel calibration samples**
Two “comb-shaped” samples made of nickel are used as standards for all measured thermal transport properties. Refer to Section 2.2.3 for more information on the nickel samples.
- **Gold-plated copper samples**
Two similar comb-shaped samples of copper are provided as thermal shunts to help provide an isothermal environment while calibrating shoe assemblies.
- **Calibration fixture**
This fixture is used in conjunction with calibration software and allows the heater and thermometer shoes to be calibrated.
- **Consumable items**
These items consist of a sampler of gold-plated copper leads, including both wires and disks; a sample of two types of epoxies for mounting leads; and a small tube of Apiezon H Grease to increase thermal contact between the leads and shoes or the coldfoot. Chapter 4 contains more detailed information about mounting leads to samples.
- **User tools**
These tools include small slotted and Phillips screwdrivers for sample mounting, and an extractor tool to remove the electrical connector plug ends of the shoe assemblies from the puck. This extractor tool holds the connector plug by sliding into the grooves on each side of the plug.

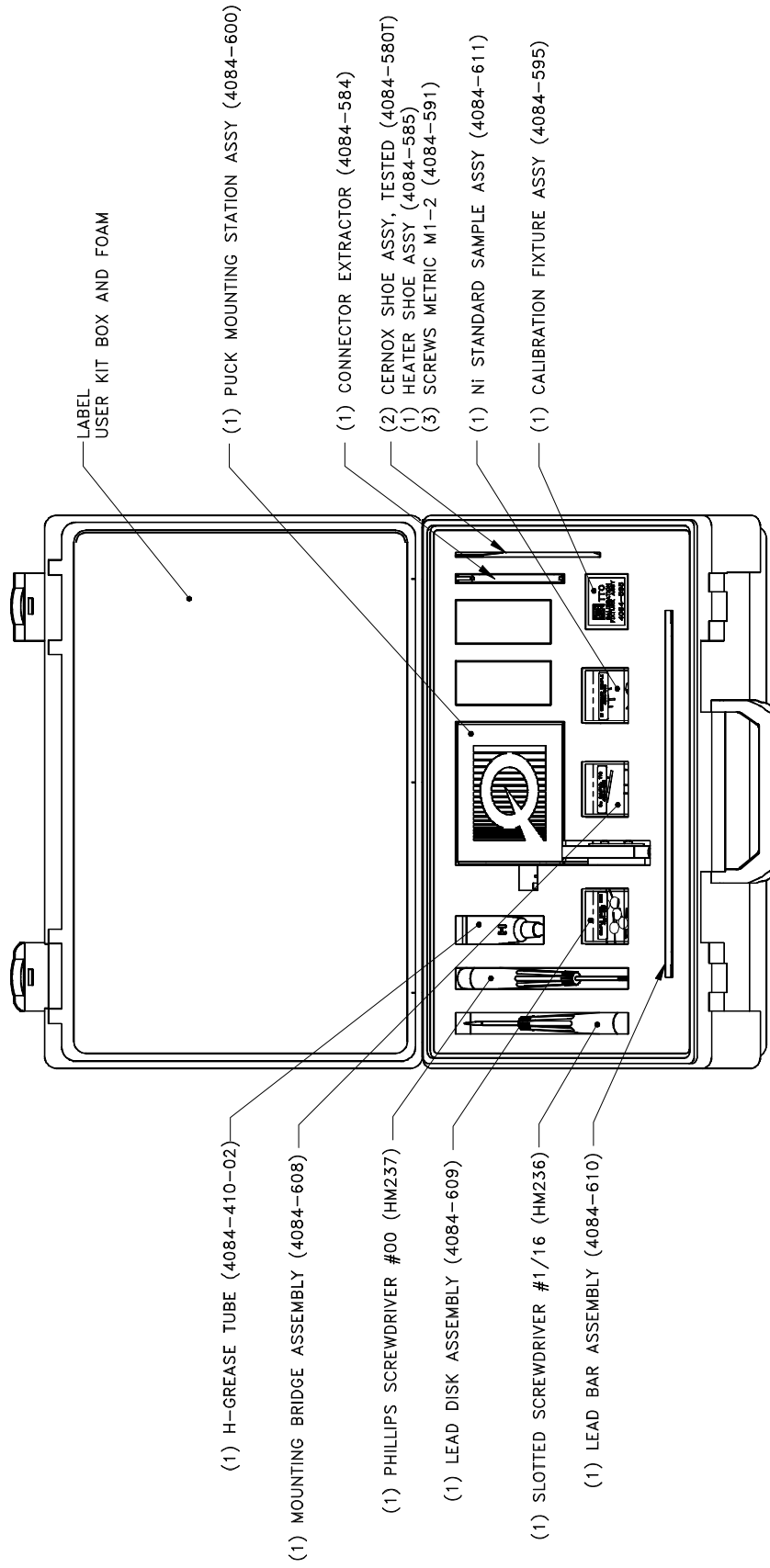


Figure 2-3. Thermal Transport Option User's Kit

2.2.3 Nickel Calibration Samples

Two samples of nickel metal (grade 201) are supplied in the user's kit in the form of thin plates stamped in a four-probe "comb" configuration (see Section 4.3) and can be used as references and calibration verification for all standard thermal transport measurements: thermal conductivity κ , Seebeck coefficient α , and electrical resistivity ρ . Variations in the geometrical factor A/l on the order of $\pm 5\%$ are to be expected from sample to sample, which will be reflected in the κ and ρ data. Table 2-1 lists standard dimensions for the Ni standard. It is also recommended that the Ni standard be mounted on the puck in the vertical configuration in order to avoid touching the radiation shield.

Table 2-1. Recommended Sample Parameters for Nickel Calibration Samples

PARAMETER	VALUE
Cross-sectional area	0.32 mm ²
Length	8.3 mm
Surface area	35 mm ²
Emissivity	0.5

2.2.4 WaveROM EPROM

The DSP board that is used with the ACMS and ACT options contains a square ROM chip that holds waveform tables for the excitation current generated by the DSP. The TTO system requires that a new waveform table—that of a square-wave pulse—be added to this library. All new AC boards include this new waveROM chip, but some customers with older boards must install the new chip in order to use the TTO system. If you are one of these customers, a new waveROM and a PLCC chip extraction tool (AC board ROM upgrade kit) have been sent to you so that you can swap out the old chip for the new one. Refer to Section 2.2.4.1.

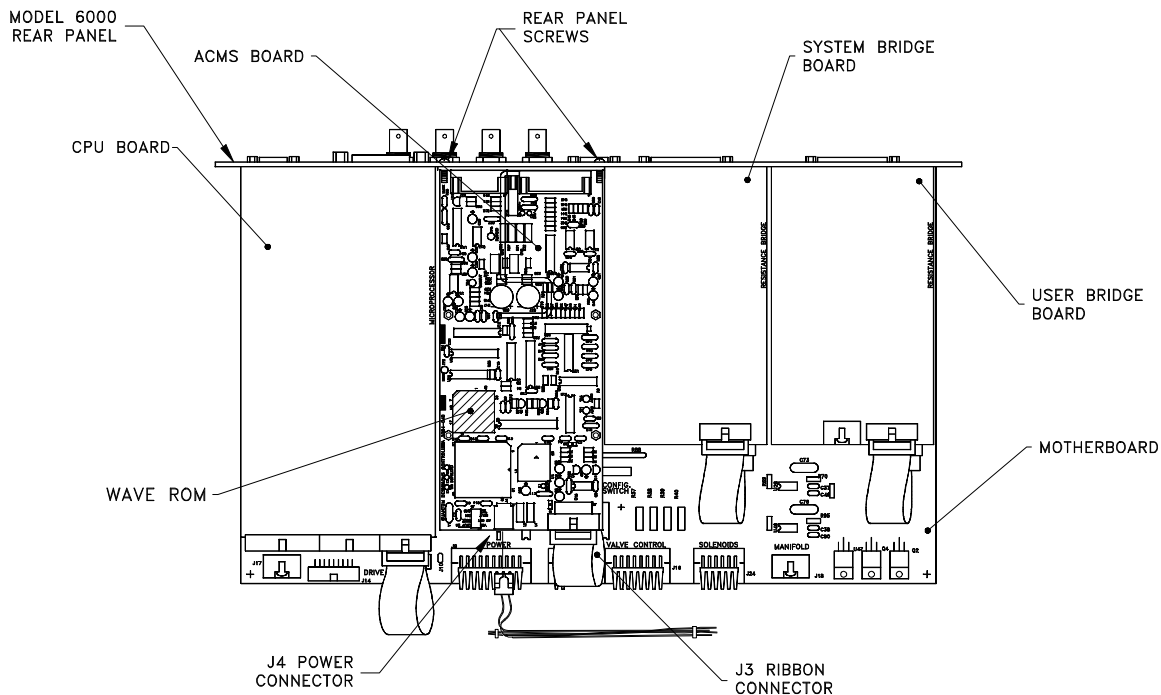


Figure 2-4. WaveROM EPROM on AC Board in Model 6000 PPMS Controller

2.2.4.1 REPLACING THE WAVEROM CHIP

To replace the waveROM chip on the AC board, first locate the upgrade kit, which contains the PLCC chip extraction tool and the new ROM in a small plastic box. Remove the lid of the Model 6000 so that you can access the AC board (note that the AC board may reside in the Model 6500 Option Controller instead). Turn off the Model 6000 and extract the old waveROM by inserting the hooks of the extraction tool in the two slots on opposite corners of the ROM housing and gently squeezing until the chip lifts out. To insert the new ROM, note that the upper left corner (as viewed in Figure 2-4) is notched, and the upper side has the label attached. Press down, applying firm and even pressure to the chip until it is seated in the housing.

2.2.5 Thermal Transport Connection Cable

The Thermal Transport connection cable (part number 3084-582) connects the sample to the Model 7100 AC Transport Controller and to the user bridge board that is in the Model 6000 PPMS Controller. Two separate shielded cables on the connection cable plug into the Model 7100. These separate shielded cables split the sample signal and excitation signal in order to help prevent sample signal distortion by the excitation signal.

Labels on the cable's connectors (Figure 2-5) identify the ports for those connectors. You should also refer to Figure A-1, the "Thermal Transport Option Connection Diagram."

- The 14-pin Lemo connector plugs into the gray, color-coded port on the PPMS probe head.
- The connector labeled "J1 (P1) User Bridge" plugs into the "P1–User Bridge" port on the Model 6000.
- The connector labeled "J2 (P1) Sample Current Out" plugs into the "P1–Sample Current Out" port on the Model 7100.
- The connector labeled "J5 (P5) Sample Voltage In" plugs into the "P5–Sample Voltage In" port on the Model 7100.

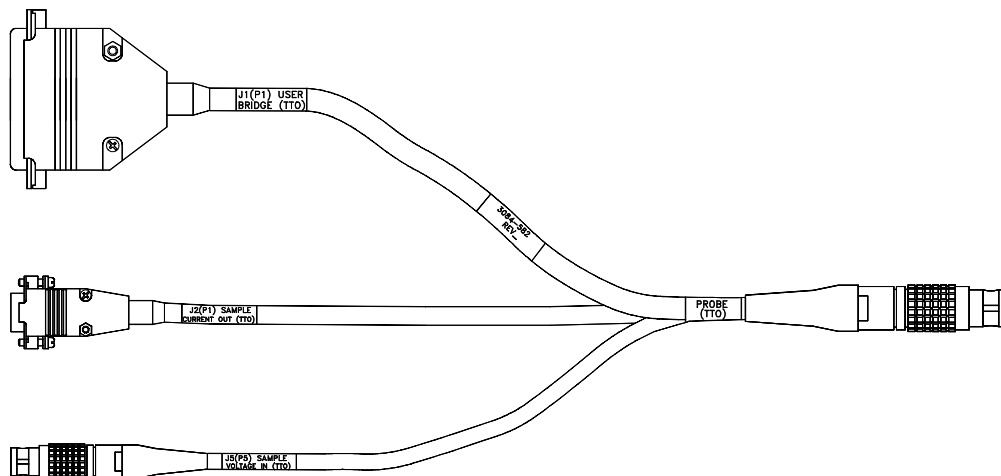


Figure 2-5. Thermal Transport Connection Cable

2.2.6 User Bridge Board

The TTO system employs the user bridge board to read the hot and cold Cernox thermometer shoe assemblies that are on the sample. The user bridge board is in the Model 6000 (see Figure 2-4). Detailed information about the user bridge board is in the *Physical Property Measurement System: Resistivity Option User's Manual*.

2.3 ACT Hardware

The TTO system uses hardware for the AC Transport Measurement System (ACT) option in order to generate the heat pulse and read back the sample thermal voltages in the thermal measurements, and to make the four-probe resistivity measurement on the sample.

2.3.1 Model 7100 AC Transport Controller

The driver board in the Model 7100 AC Transport Controller excites the sample by receiving and amplifying the signal from the AC board's digital signal processor (DSP). The preamp board in the Model 7100 detects the sample signal and sends the signal back to the DSP so the DSP can process the signal. The *Physical Property Measurement System: AC Transport Option User's Manual* discusses the components and operating modes of the Model 7100 in more detail.

CAUTION! The Model 7100 provides as much as 200 mA of current when being controlled by the TTO system. Although this is lower than the hardware limit of 2 A, this current can still damage samples in the current path. Use only currents that can be safely handled by all hardware and samples in the circuit.

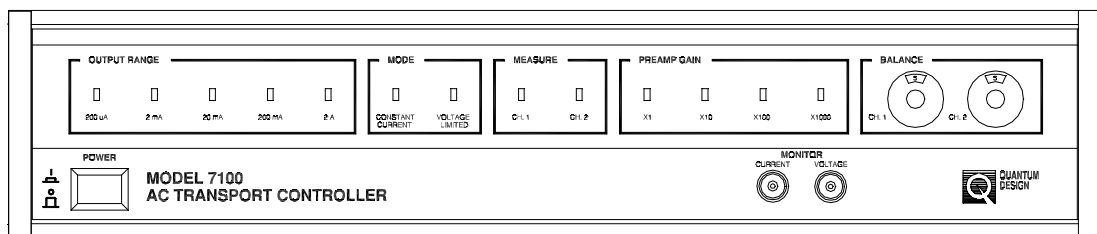


Figure 2-6. Front Panel on Model 7100 AC Transport Controller

2.3.2 AC Board

The AC board is installed in the Model 6000 PPMS Controller and is located behind the “P3–Option” port, which is the port connecting the Model 6000 to the Model 7100. The waveROM EPROM (Section 2.2.4) plugs into the AC board. The AC board includes a DSP, digital-to-analog converter (DAC), current drivers, and other control electronics that are necessary to synthesize excitation signals and process sample response signals. The DSP provides the excitation waveform and processes the sample signal.

2.4 High-Vacuum Hardware

The High-Vacuum option, which operates in conjunction with the TTO system, reduces the amount of gas in the sample chamber and thus minimizes stray thermal conduction from the heated sample. The TTO system works with either the Turbo Pump High-Vacuum option or the Cryopump High-Vacuum option. The details of the Turbo Pump and Cryopump high-vacuum systems are contained in the *Physical Property Measurement System: Turbo Pump High-Vacuum Option User's Manual* and *Physical Property Measurement System: Cryopump High-Vacuum Option User's Manual*, respectively.

2.4.1 Contact Baffle

An integral part of either the Turbo Pump or the Cryopump High-Vacuum system is the contact baffle assembly. The contact baffle makes thermal contact with the isothermal region of the sample chamber, which is just above the puck. The thermal contact between the contact baffle and the isothermal region helps create a more uniform thermal environment for the puck by causing the contact baffle to be at the same temperature as the chamber walls that are near the puck. This is important when high vacuum is enabled; high vacuum reduces the amount of thermal exchange gas in the sample chamber.

You insert the contact baffle into the brass fitting that is at the bottom of the baffle assembly (see Figure 2-8B). To help safeguard the contact baffle, use it only when you are using the High-Vacuum option. Handle the contact baffle with care, and avoid touching the delicate outer contact fingers. The charcoal holder is used to achieve the best vacuum at low temperatures and should always be screwed onto the bottom of the contact baffle when in the high-vacuum state. However, when performing a temperature calibration of any sample hardware, such as TTO shoe assemblies, the charcoal holder should be removed to ensure that adequate thermal exchange gas remains in the sample chamber at low temperature.

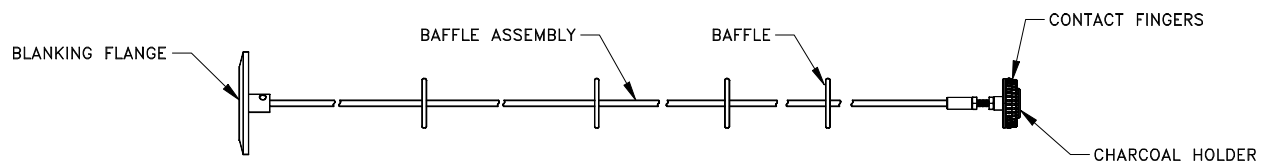


Figure 2-7. Baffle Assembly with Contact Baffle

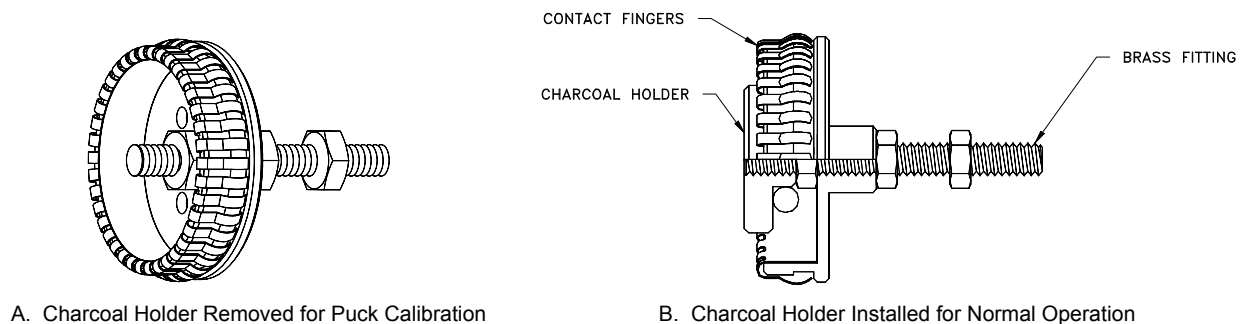


Figure 2-8. Close-up View of Contact Fingers and Charcoal Holder on Contact Baffle Assembly

2.5 Calibrating New Shoe Assemblies

A spare set of uncalibrated shoe assemblies (two thermometers and one heater) is included in the TTO user's kit. You can easily calibrate the shoe assemblies by using the calibration wizard (see Figure 2-10) that is accessed in the **Advanced** tab of the Thermal Transport control center. To calibrate new shoe assemblies, the calibration fixture (3084-576) must first be plugged into the Thermal Transport puck as shown in Figure 2-9.



The vertical plate that is usually mounted between the sample and the plugs for the shoe assemblies must be removed before plugging in the calibration fixture. Unscrew the two Phillips-head screws at the base of the plate only enough to remove the plate, and then retighten the screws to hold the PC board. Use caution so that you do not strain the wiring on the bottom side of the PC board: do not lift or turn the board or pinch any wires when retightening the screws.

If a heater shoe is being calibrated, plug it into the left-hand socket (the socket closest to the marking "PCB 3084-576" –see Figure 2-9). If calibrating thermometer shoe assemblies, plug them into the other two sockets, with "Thermometer A" in the middle socket and "Thermometer B" in the right-hand socket. Note that wiring in the shoe assemblies is symmetric, so plugging in the connectors in either of the two possible orientations will make the proper electrical connections.

Next, locate a gold-plated copper calibration sample from the TTO User's Kit. Bend the leads on the sample so that you can mount it as shown in Figure 2-9. Then mount the copper sample to the cold foot and mount the copper shoes to the sample. When mounting the heater shoe, make sure the copper sample does not touch the solder pad of the heater resistor, which could cause an electrical short.

Note the serial numbers of each shoe assembly. Then screw the shield onto the TTO puck. Unscrew the shield cap and make sure none of the copper shoes are touching any part of the puck or the shield. Replace the shield cap; then insert the puck into the PPMS. Remove the charcoal carrier on the contact baffle assembly (see Section 2.4.1) to ensure exchange gas is not cryopumped at low temperatures. Place the baffle set inside the sample chamber. Then purge and seal the sample chamber.

In the calibration wizard window, check the box for the thermometers and/or heaters you wish to calibrate and enter their serial numbers. The default temperature range should be 1.8 to 400 K. The heater parameters have been selected to optimize the signal for the 2k Ω heater resistors supplied. Press the "Start" button to begin calibration, which will last approximately 16 hours.

After calibration is complete, you must make the appropriate changes to TTO initialization file TTO.INI if you wish to use the newly calibrated shoe assemblies immediately. See Section 3.2.

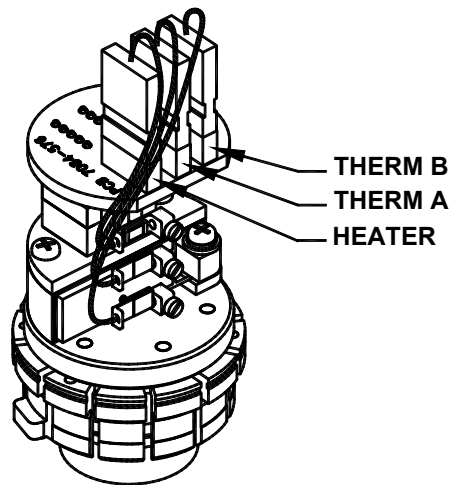


Figure 2-9. Calibration Fixture Plugged into TTO Puck and Illustrating Sockets for Each Shoe Assembly

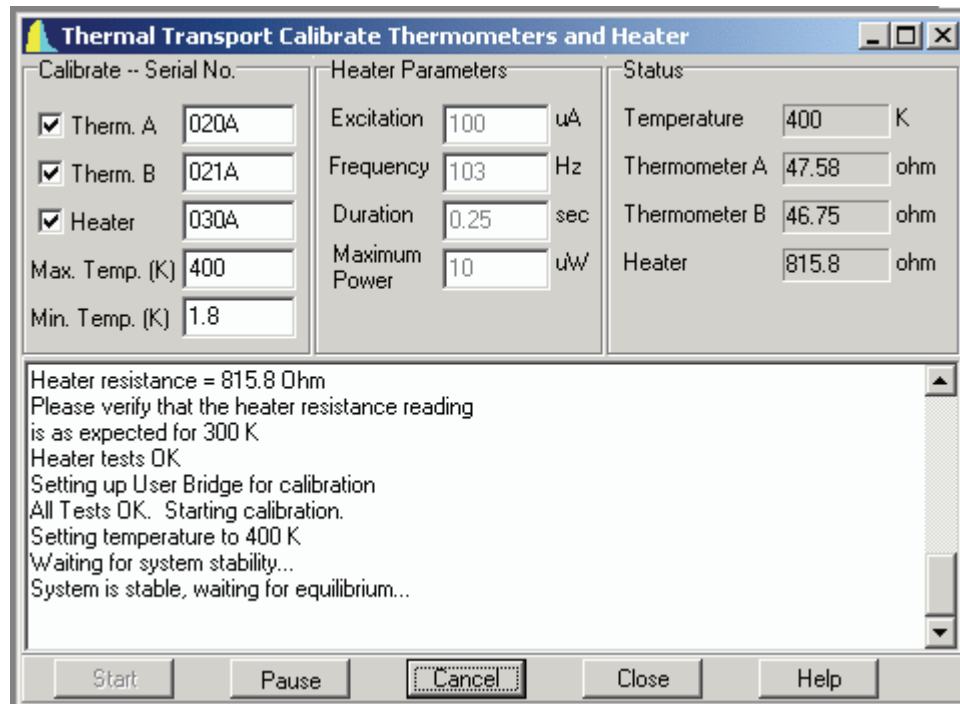


Figure 2-10. Thermal Transport Calibrate Thermometers and Heater Wizard

Software

3.1 Introduction

This chapter contains the following information:

- Section 3.2 presents an overview of the TTO system software.
- Section 3.3 discusses the Thermal Transport control center.
- Section 3.4 discusses the TTO data files.
- Section 3.5 explains how to examine data saved to a TTO data file.

3.2 Overview of Thermal Transport Software

The TTO software module is integrated into the Quantum Design PPMS MultiVu environment. Version 1.1.6 or greater of the PPMS MultiVu software is required to install the TTO software. For software installation instructions, see Appendix A.

TTO is designed to be used by both experts and newcomers to the study of thermal transport properties. To accommodate this spectrum of users, the software interface is multi-leveled so that beginners can easily set up and perform measurements at the top level while experts can choose to navigate into submenus in order to adjust various parameters or customize measurements.

TTO measurements can be run either interactively at the dialog or in a sequence program. While the TTO software is active in PPMS MultiVu, the command **TTO Measure** appears in the sequence command bar under the **Measurement Commands** heading. This command is used to start, stop, or change measurement parameters within a sequence file. Users who are familiar with measurement sequence commands for other Quantum Design options will find an important difference in the use of the **TTO Measure** command compared to other options, where sequence execution is typically paused until a measurement command is completed. This principle is not appropriate for continuous TTO measurements because these can be performed while a system parameter such as the temperature is swept slowly. Therefore, the **TTO Continuous Measure** command initiates measurements and the sequence execution immediately continues. TTO measurements are taken until the **TTO Stop** sequence command is issued. The TTO single measurement sequence commands are more similar to the traditional measurement commands. During these measurements, sequence execution is paused until the measurement is completed because single measurements operate on the condition that system parameters are stable during the measurement.

The data file for TTO measurements (*sample.dat*, where *sample* is the file name you select) contains the results for each measurement that was completed successfully. The raw data from all measurements can also be saved in a separate file (*sample.raw*).

As soon as the TTO software is activated in PPMS MultiVu, the **Thermal Transport Log** window (Figure 3-1) opens and indicates which thermometer and heater calibration files will be used. These settings, as well as several other parameters, can be edited in the *Tto.ini* initialization file located in the *QdPpms\ThermalTransport\System* directory. The thermometer and heater calibration files are located in the *QdPpms\ThermalTransport\Calibration* directory. The TTO log can be viewed by using the **View>TTO Status Log** menu command.

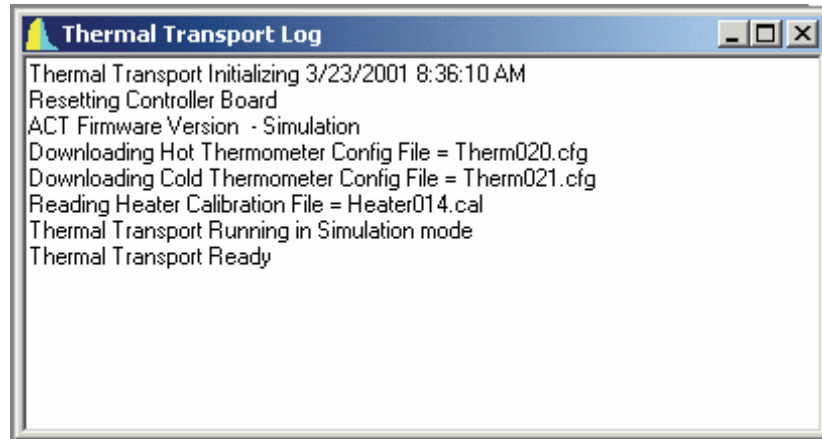


Figure 3-1. **Thermal Transport Log** Window

3.2.1 Measurement Units

Thermal conductivity κ is typically stated in either units of watt/(meter-kelvin), written W/m-K, or in mW/cm-K. In the TTO software, the units of W/m-K are used for thermal conductivity. The Seebeck coefficient is expressed most conveniently in $\mu\text{V/K}$, because in many electrical conductors $\alpha \sim \mu\text{V/K}$ at room temperature. The electrical resistivity ρ is stated in units of ohm-meter ($\Omega\text{-m}$). The thermoelectric figure of merit $ZT = \alpha^2 T / \rho \kappa$ is dimensionless when MKS units are chosen for each quantity; that is, $\alpha[\text{V/K}]$, $T[\text{K}]$, $\rho[\Omega\text{-m}]$, and $\kappa[\text{W/m-K}]$.

3.3 Thermal Transport Control Center

The TTO software module has a control center that includes all frequently selected Thermal Transport commands. With its easy-to-use tab format and software prompts, the control center makes basic system operations, such as installing samples, creating data files, and setting up and running immediate-mode measurements, more natural and convenient. The Thermal Transport control center opens as soon as the Thermal Transport option is activated in PPMS MultiVu, and although it may be minimized, does not close until the option is deactivated. Figures 3-2 through 3-6 illustrate the tabs in the Thermal Transport control center.

3.3.1 Control Center Tabs

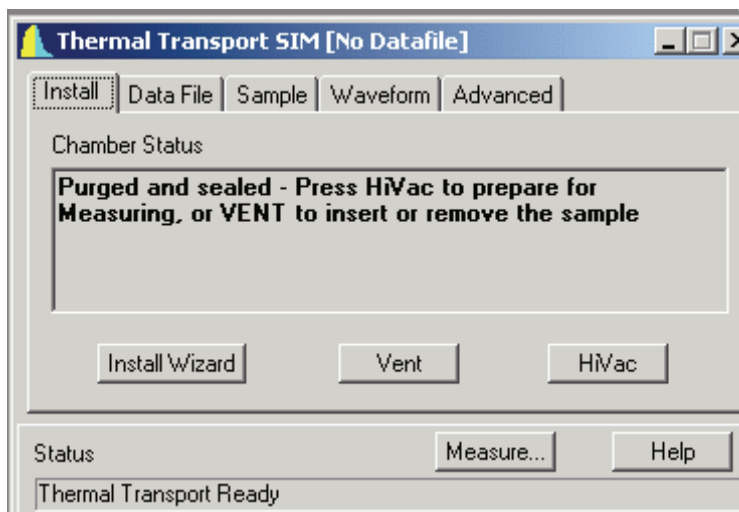


Figure 3-2. Control Center **Install** Tab

The **Install** tab automatically opens when the software starts, and it assists you in sample installation or removal by providing sample chamber **Vent** and **HiVac** buttons and by also providing an install wizard with more extensive, step-by-step instructions for setting up a measurement.

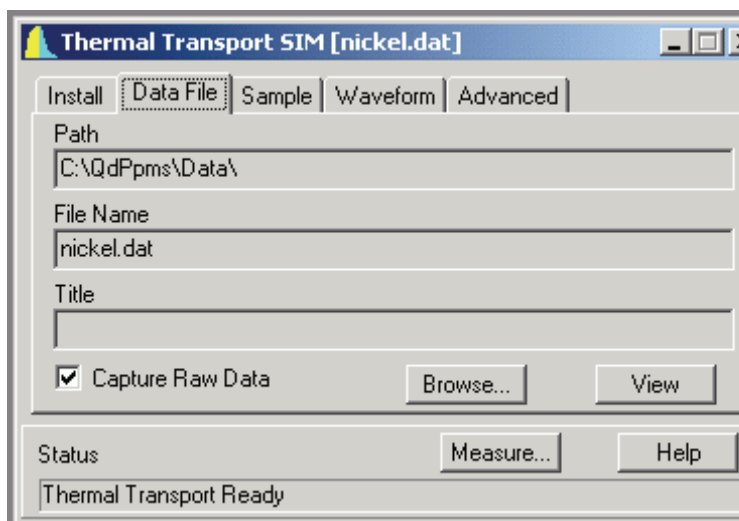
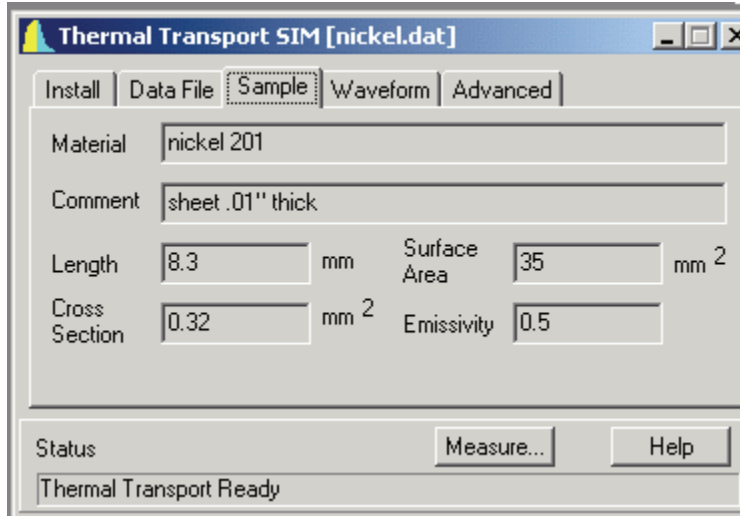


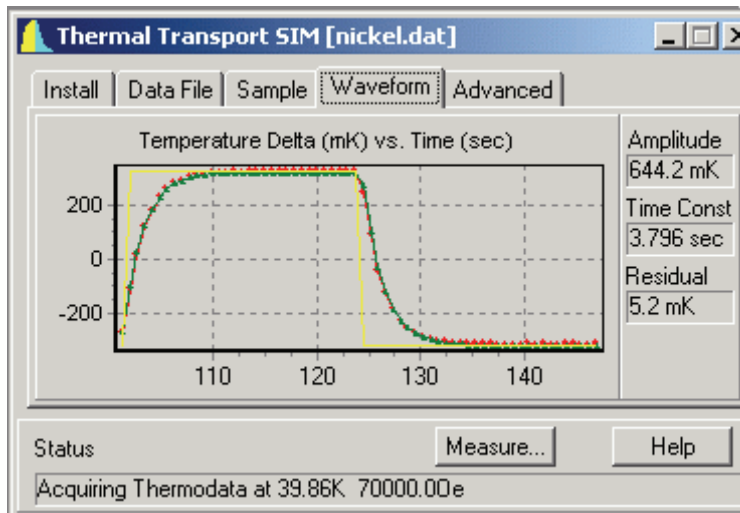
Figure 3-3. Control Center **Data File** Tab

The **Data File** tab lets you open a new TTO data file or append to an existing TTO data file. You can also use the tab to view data and select whether raw thermal transport data is saved in a .raw file. See Section 3.4 for information on TTO data file formats.



The **Sample** tab displays the sample name, comments, geometry and radiation estimates for the currently open data file. These parameters can be changed only by opening a new file in the **Data File** tab. Chapter 4 has more information on how to mount sample leads and estimate properties such as the sample's infrared emissivity.

Figure 3-4. Control Center **Sample** Tab



The time traces of the most recent raw data, along with the fitted curves, are displayed in the **Waveform** tab. As a visual aid, the heater pulse is shown schematically in the **Waveform** tab as a yellow square wave in both the thermal conductivity and Seebeck graphs.

Figure 3-5. Control Center **Waveform** Tab

Clicking the right mouse button inside the graph window in the **Waveform** tab opens a menu that allows selection of thermal conductivity, Seebeck coefficient, or resistivity results, and whether to plot the fitted curve along with raw data. The title of the graph indicates which data is being displayed. For thermal conductivity data, “Temperature Delta” refers to the difference between hot and cold thermometers, while for Seebeck measurements “Seebeck Voltage” refers to the voltage difference between the hot and cold shoes.

To the right of the graph are listed three parameters that briefly summarize the curve fitting results: the total amplitude obtained by the curve-fitting routine (equivalent to $\Delta \text{Temp.}$ or Seebeck Volt. in the data file), the long time constant in the measurement (that is, τ_1 —this is not relevant to resistivity data), and the residual of the curve fit (that is, the error estimate for the reported total amplitude). Section 1.5 contains more information on the AC measurements and error estimation in TTO data.

In the **Waveform** tab, you can also zoom to examine details of the data by dragging the mouse from the upper left to the lower right corner of the graph while holding down the left mouse button. To zoom out, drag the mouse in the opposite direction while holding down the button, or select **Zoom All** in the pull-down menu, which is accessed by clicking the right mouse button in the graph.

The **Advanced** tab includes a software wizard that allows you to calibrate new TTO shoe assemblies (heater and/or thermometers) in the PPMS. The calibration fixture (3084-576) from the user’s kit is used in conjunction with this software wizard. The **Restart...** button is used if you previously cancelled a calibration and want to restart. Refer to Section 2.5 for more information on calibrating shoe assemblies.

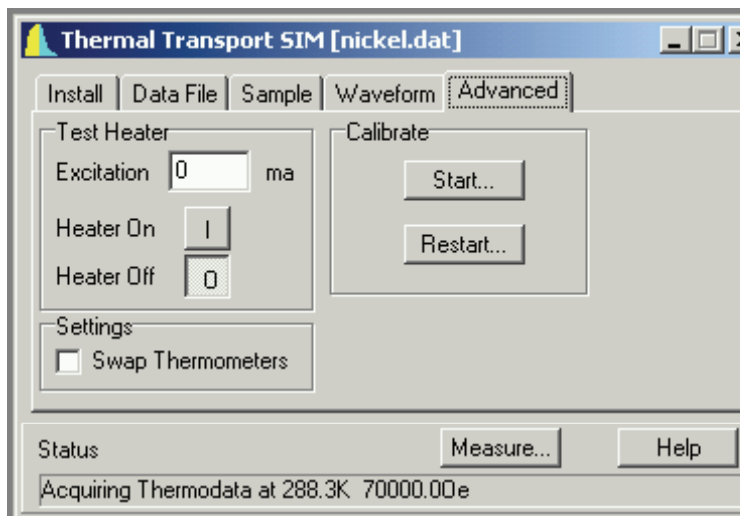


Figure 3-6. Control Center **Advanced** Tab

Other features on the **Advanced** tab include a heater test in which you select the desired heater current to apply, as well as an option to swap the software’s assignment of hot and cold thermometers.

3.3.2 Measurement Menu

Selecting the **Measure...** button at the bottom of the Thermal Transport control center opens the **Thermal Transport Measurement** dialog box, which allows you to run immediate-mode TTO measurements without having to write a sequence file. Three tabs—**Settings**, **Thermal**, and **Resistivity**, shown in Figures 3-7, 3-8, and 3-9—are immediately visible in the **Thermal Transport Measurement** dialog.

In the **Settings** tab (Figure 3-7) you determine basic settings for all measurements. First you select which of the four thermal transport quantities are to be measured. Note that checking **Figure of Merit (ZT)** automatically selects all measurements because they are all required to assess ZT. You have the

option to save “marginal” results, which would otherwise be discarded, to the data file. Marginal measurements are defined as those for which the software was able to determine a quantity, but the regression errors of the curve fits were between 50% and 200% (the measurement is considered failed if the regression is higher than 200%). The **Discard First N Results** check box is included because the first several measurements often have large errors due to the measurement parameters not being optimized. Choosing $N = 3$ is typically adequate, if you choose to discard any results. In the **Next Measurement** box you can specify the period and heater power for the next thermal measurement cycle, even after measurements have started. Note that any changes made in this box are saved only if the **Set** button is selected. Selecting **Clear** restores the values that were last input.

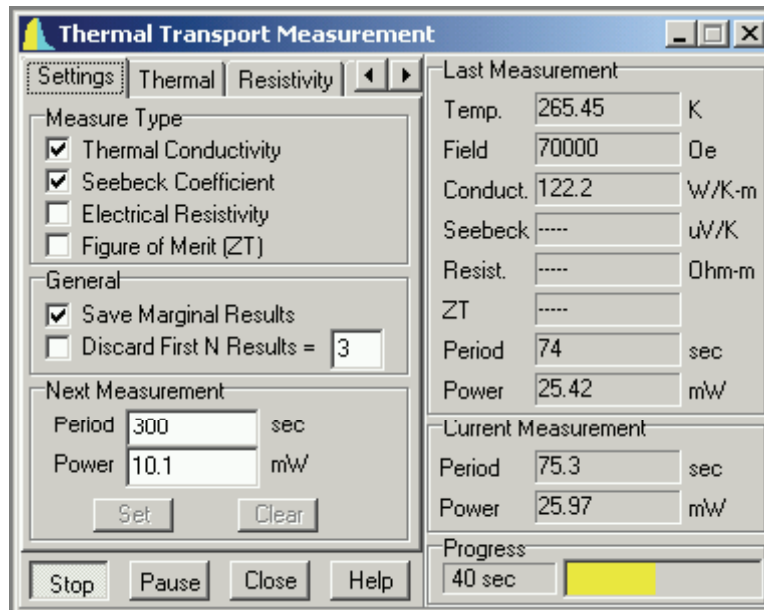


Figure 3-7. **Settings** Tab in **Thermal Transport Measurement** Dialog Box

Settings and limits for thermal measurements (thermal conductivity and Seebeck coefficient) are determined in the **Thermal** tab (Figure 3-8). These settings and limits include limits of heater period and power and expected limits of Seebeck readback voltage, as well as target amplitude for the heat pulse (expressed as percentage of sample temperature) and target value of period ratio, which is defined as measurement period divided by the time constant ($\tau\omega_1$) of the sample (see Section 3.4.3 for more information on these quantities). Any changes made in this tab are saved only if the **Set** button in the tab is selected. A ToolTip displays hardware limits for the heater power if the cursor is placed over the panels showing the heater power limits. Note that a period ratio of at least 8 is recommended because choosing a value lower than this has been shown to produce artifacts in the thermal transport measurements due to insufficient data for the software model.

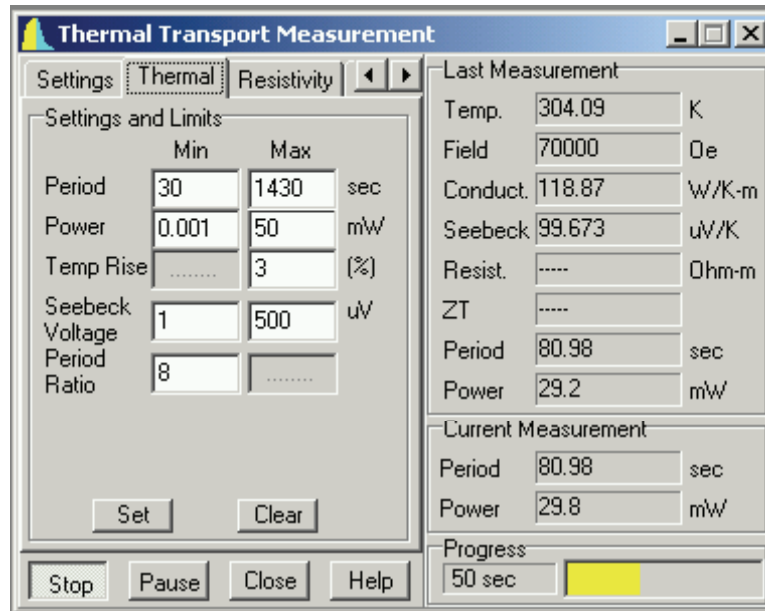


Figure 3-8. Thermal Tab in Thermal Transport Measurement Dialog Box

Due to the complexity of making an adaptive resistivity measurement, a separate tab is devoted to resistivity (Figure 3-9) so that you can have maximum freedom in setting limits such as min/max excitation and min/max frequency, as well as measurement duration. The measurement excitation frequency is allowed to vary so that a 90% resistive (that is, in-phase) signal is obtained. Autoranging parameters may also be changed, with the default being **Sticky Autorange**. Changes made in this tab are saved only if the **Set** button in the tab is selected. See the *Physical Property Measurement System: AC Transport Option User's Manual* for more information on resistivity measurement parameters.

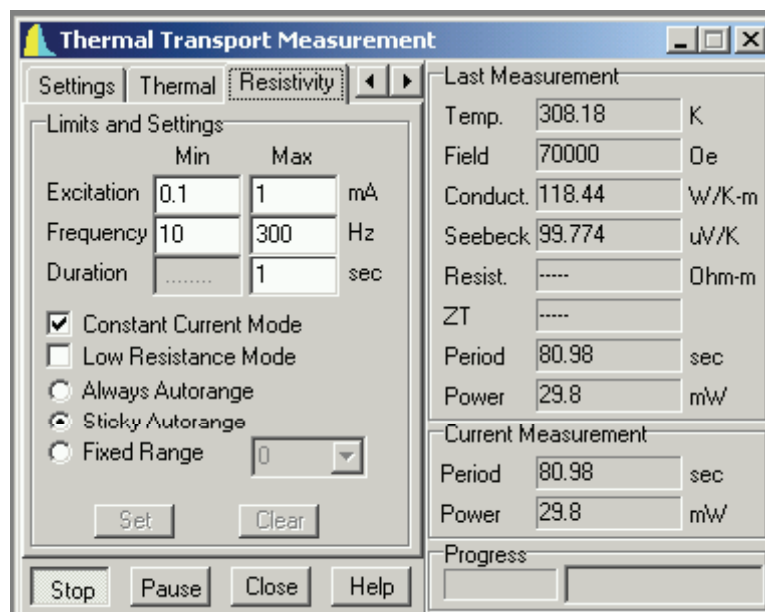
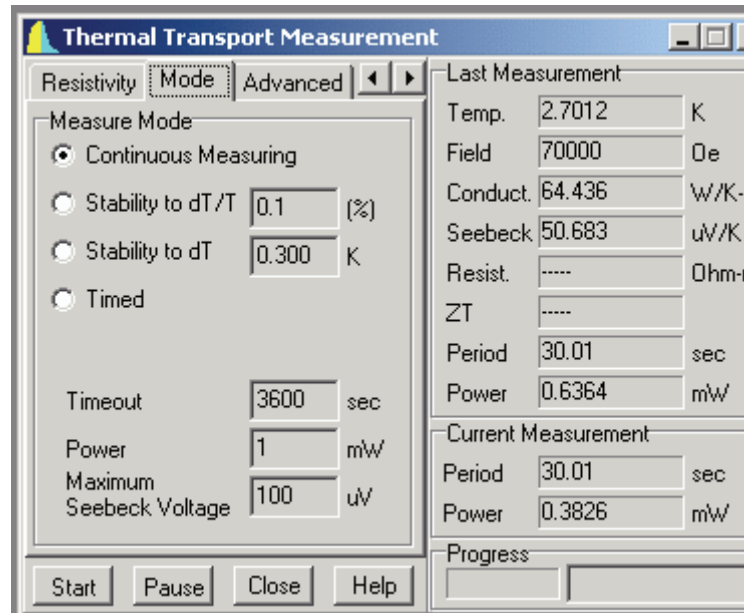


Figure 3-9. Resistivity Tab in Thermal Transport Measurement Dialog Box

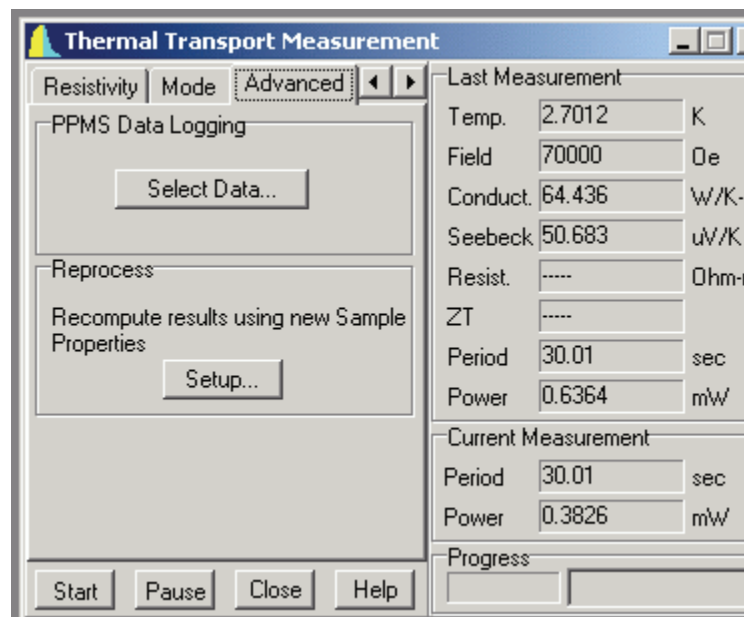
3.3.2.1 OPTIONS FOR ADVANCED USERS

Two more tabs, the **Mode** tab and the **Advanced** tab, are made visible in the **Thermal Transport Measurement** dialog box by clicking on the right-pointing arrow that is next to the tab names at the top of the dialog box. Clicking on the left-pointing arrow hides the **Mode** and the **Advanced** tabs. Generally, these two tabs are of interest only to advanced users.



You use the **Mode** tab to select whether to take continuous measurements (default) or single, steady-state measurements. There are two modes of single measurement: stability or timed. Section 1.3 describes the measurement modes in more detail.

Figure 3-10. **Mode** Tab in **Thermal Transport Measurement** Dialog Box



The PPMS data logging dialog can be accessed from the **Advanced** tab in the event that you would like to select which PPMS status items are written to the TTO data file. Table 3-1 on the following page describes the parameters that can be monitored on the PPMS data logging dialog. The **Reprocess** command allows you to recompute thermal transport results from an existing TTO data file (*.dat) by changing the sample's estimated geometry and emissivity, and the command allows you to write these results out to a new data file.

Figure 3-11. **Advanced** Tab in **Thermal Transport Measurement** Dialog Box

Table 3-1. PPMS system data items that can be saved to the TTO measurement data file. Items in bold and all capital letters are always written to the data file.

ITEM	DEFINITION
GENERAL STATUS	General system status, coded. Appendix B explains how to interpret status code.
TEMPERATURE	System temperature, measured at base of sample chamber.
MAGNETIC FIELD	Magnetic field.
Sample Position	For use with rotator probes*.
SAMPLE PRESSURE	Pressure in sample chamber, measured in torr.
Digital Inputs	Eight-bit status of selected inputs.
Dr Ch 1–2 Current	Current delivered by selected driver output channel.
Dr Ch 1–2 Power	Power delivered by selected driver output channel.
Brg Ch 1–4 Resistance	Resistance of selected user bridge channel.
Brg Ch 1–4 Excitation	Excitation current of selected user bridge channel.
Sig Ch 1–2 Input Voltage	Input voltage for selected signal channel.
Map 20–29 MAP 21–22	User-designated data items. Reserved for hot and cold sample thermometers.

* Rotator probes are not currently available for use with TTO.

3.3.3 System Status

Features in the Thermal Transport control center provide constant updates of TTO status.

- The **Status** bar at the bottom of the Thermal Transport control center succinctly describes the progress of an on-going measurement and summarizes the results of the last measurement. Color-coded warning and error messages in the **Status** bar alert you to possible problems. Warning messages appear on a yellow background. Error messages appear on a red background.
- The **Waveform** tab in the Thermal Transport control center (Figure 3-5) displays raw data waveforms and curve fits for any of the measurements, and the tab indicates the fitting parameters of signal amplitude, time constant τ_1 (where relevant) and the residual for the fit.
- The measurement **Progress** bar and countdown timer in the bottom right corner of the **Thermal Transport Measurement** dialog box indicate the time remaining before the measurement is complete. Refer to Figure 3-7. In the **Progress** bar, yellow indicates the heater “on” state while blue indicates “off.”
- The right side of the **Thermal Transport Measurement** dialog box summarizes the most recent measurement and the parameters (period and power) used for the current measurement. Refer to Figure 3-8. Note that if the cursor is placed over the displayed results, a ToolTip shows the estimated error for the results.
- The **Thermal Transport Log** window (Figure 3-1) keeps a record of all messages that appear in the Thermal Transport control center **Status** bar. The TTO status log is saved in the file `TtoLog.txt`, which is in the directory `C:\QdPpms\ThermalTransport\LogFiles`.

3.4 Thermal Transport Data Files

Thermal Transport measurement data is stored in measurement data files and raw measurement data files. Measurement data files, which have a `.dat` extension, store relevant sample measurement data and selected system data for any number of measurements taken by any of the Thermal Transport measurement types. Raw measurement data files, which have a `.raw` extension, store raw temperature versus time (thermal conductivity) and voltage versus time (Seebeck and resistivity) data as well as the software's fits to the data. Raw data is saved only if the **Capture Raw Data** check box in the **Data File** tab (Figure 3-3) is selected. The system data items that may be recorded are user configurable (Section 3.4.2).

The results from measurements, whether made in continuous or single mode, are automatically saved if a data file is open. Measurement and system data is appended to the selected data files and is never overwritten. The **Data File** tab in the Thermal Transport control center identifies the selected measurement data file and raw measurement data file and includes a **Browse** command button that enables data file selection and creation. The name of the selected measurement data file is also displayed in the title bar of the Thermal Transport control center.

3.4.1 Saving Raw Thermal Transport Data

Raw thermal transport data is saved to a separate raw measurement data file that has the identical base name as the measurement data file, but a `.raw` file extension instead of a `.dat` extension. Saving the raw data can be useful when you are deciding which measurement parameters to use or if you are concerned about signal quality. A clean, single-wavelength sine wave is optimal in the case of AC resistivity measurements. In the cases of thermal conductivity and Seebeck measurements, the fitted model curve can be plotted along with the raw data so that you can easily assess the quality of the results. However, saving raw data creates very large data files.

Raw thermal transport data is saved only if a measurement data file is selected *and* the software is prompted to capture raw data. Enabling the **Capture Raw Data** check box in the Thermal Transport control center's **Data File** tab (Figure 3-3) prompts the software to save raw data. By default, this check box is *not* selected.

3.4.2 Data File Header

The data file header contains file and sample property information that is defined when the data file is created. Information written to the data file header cannot be subsequently changed in PPMS MultiVu. The file information that can be written to the header consists of the title assigned to the graph view of the data file. The sample property information consists of the sample dimensions and emissivity. User comments can also be written to the header. All this information appears in the `INFO` declarations of the header. The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* contains more information on data file headers.

When you create a data file, the software prompts you to enter the physical dimensions and an estimate of the emissivity for the sample whose measurement data will be saved to the file. To enable you to do this, the data entry fields in the Thermal Transport control center's **Sample** tab are enabled. As soon as you define the sample properties and select **OK**, all data entry fields in the **Sample** tab (Figure 3-4) are disabled again.



Because the data file header identifies a particular sample, changing samples without changing data files can destroy the validity of the data in the file. Therefore, you are encouraged to use the automated routines in the Thermal Transport control center. These automated routines prompt you for new data file(s) and new sample information after you install a different sample.

3.4.3 Format of Measurement Data Files

Table 3-2 lists and describes all data columns in the Thermal Transport data file. Section 5.3 provides more information on some of these fields.

Table 3-2. Fields in Thermal Transport Measurement Data File

ITEM	DEFINITION
Time Stamp	Time of measurement data point, expressed in minutes or seconds, and as an absolute time or relative to the start time of the data file.
Status (code)	PPMS system status. Identical to General Status in Table 3-1.
Error (code)	TTO error code. Appendix B describes how to interpret the code.
Magnetic Field (Oe)	Magnetic field.
Sample Temp. (K)	Average sample temperature during measurement.
Conductivity (W/K-m)	Sample thermal conductivity.
Cond. Std. Dev.	Error (standard deviation) in thermal conductivity measurement.
Seebeck Coef. (uV/K)	Sample Seebeck coefficient in units of $\mu\text{V}/\text{K}$.
Seebeck Std. Dev.	Error in Seebeck coefficient measurement.
Resistivity (Ohm-m)	Sample resistivity.
Resist. Std. Dev.	Error in resistivity measurement.
Figure of Merit [ZT]	Dimensionless thermoelectric figure of merit ZT.
Merit Std. Dev.	Error in ZT measurement.
Delta Temp. (K)	Extrapolated (asymptotic) temperature drop ΔT across heated sample.
Conductance (W/K)	Net thermal conductance of sample. See Section 1.5.5.
Raw Conductance (W/K)	Raw thermal conductance, that is, (Heater Power)/(Delta Temp.).
Seebeck Volt. (uV)	Extrapolated (asymptotic) Seebeck ΔV across heated sample.
Resistance (Ohm)	Sample resistance.

(continues)

Table 3-2. Fields in Thermal Transport Measurement Data File (Continued)

ITEM	DEFINITION
Min. Temp. (K)	Minimum temperature at either hot or cold thermometer during measurement.
Max. Temp. (K)	Maximum temperature at either hot or cold thermometer during measurement.
Temp. Rise (K)	Rise in temperature of the hot thermometer due to the applied heat pulse. Should be close to user-requested value set in Thermal tab.
Req. Htr. Power (W)	Requested heater power, in watts.
Heater Power (W)	Actual heater power.
Rad. Loss (W)	Estimated power loss due only to radiation from sample. See Section 1.5.5.
Cond. Pwr. (W)	Estimated net power conducted through sample.
Heater Current (mA)	Current through heater.
Res. Drive (mA)	Current drive used for resistivity measurement.
Res. Freq. (Hz)	Frequency used for resistivity measurement.
Period (sec.)	Period for heater on/off square-wave pulse.
Period Ratio	Ratio of period/tau1.
tau1 (sec.)	Long thermal time constant of sample and shoes.
tau2 (sec.)	Short thermal time constant of sample and shoes.
Seebeck Gain	Total gain (preamp and DSP) for Seebeck data point.
Resist. Gain	Total gain (preamp and DSP) for resistivity.
System Temp. (K)	PPMS block system temperature.
Sample Position (deg.)	Used with rotator probes; not used in TTO*.
Brg Ch 1–4 Resistance	Resistance of selected user bridge channel.
Brg Ch 1–4 Excitation	Excitation current of selected user bridge channel.
Sig Ch 1–2 Input Voltage	Input voltage for selected signal channel.
Digital Inputs (code)	Eight-bit status of selected inputs.
Dr Ch 1–2 Current	Current delivered by selected driver output channel.
Dr Ch 1–2 Power	Power delivered by selected driver output channel.
Pressure	Sample chamber pressure, in torr.
Map 20–29 Map 21–22	User-designated data items. Reserved for hot and cold sample thermometers.

* Rotator probes are not currently available for use with TTO.

3.4.4 Format of Raw Data Files

If the **Capture Raw Data** check box is selected in the **Data File** tab in the Thermal Transport control center (Figure 3-3), raw measurement data is recorded to a data file. For thermal conductivity and Seebeck coefficient measurements, raw data also includes results from the thermal model. For resistivity measurements, raw data includes the excitation current and signal voltage.

Table 3-3. Fields in Thermal Transport Raw File

ITEM	DEFINITION
Comment	System status and TTO software comments.
Time Stamp	Time of measurement data point, expressed in minutes or seconds, and as an absolute time or relative to the start time of the data file.
T-Hot (K)	Temperature of sample hot thermometer.
T-Cold (K)	Temperature of sample cold thermometer.
T-Sys (K)	Temperature of PPMS system thermometer.
Delta T (K)	Temperature drop across sample thermometers.
Model Delta T (K)	Curve fit of ΔT to software thermal model.
Seebeck (μV)	Raw Seebeck voltage.
Model Seebeck (μV)	Curve fit of Seebeck to software thermal model.
Res. Excit. (mA)	Excitation current from resistivity measurement.
Res. Signal (mV)	Signal voltage from resistivity measurement.

3.5 Data Examination

To examine the current TTO .dat data file in PPMS MultiVu, you can select the **View** button in the **Data File** tab in the control center. If raw data is also being saved, the raw file can be opened by using the **File>Open** menu command. The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses the graphing and data-viewing formats in detail.

The `ExportData.exe` program, located in the `QDPPMS\Tools` directory, can be used to export specific columns and portions of the data file header from any data file.

Errors encountered by the PPMS during TTO data acquisition are listed in the TTO status log (see Section 3.3.3). In addition, an **Error Count** dialog is opened if any individual TTO measurement fails. A running total of the error counts is displayed and brief one-line descriptions of the last three errors are listed in the window. If this dialog is closed, it can be accessed by using the **View>TTO Error Count** menu command. Selecting the **Reset** button will zero the displayed error totals for each measurement, but will not affect the TTO log file.

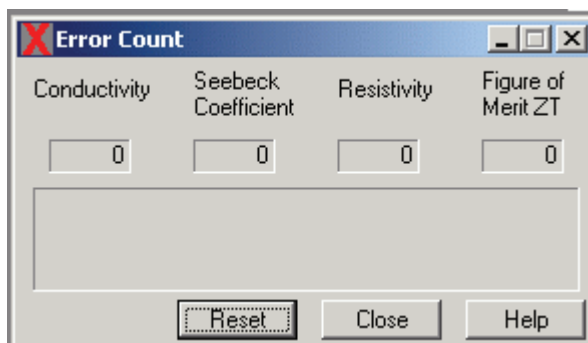


Figure 3-12. **Error Count** Dialog Box

Sample Preparation

4.1 Introduction

This chapter contains the following information:

- Section 4.2 discusses sample-mounting considerations.
- Section 4.3 discusses the two-probe and four-probe lead configurations.
- Section 4.3 explains how to check the sample contact.
- Section 4.5 explains how to use the puck-mounting station.

4.2 Sample-Mounting Considerations

Four leads must be attached to the sample in order for the TTO system to take thermal (and electrical) measurements. These four leads are a heater (and current I+), a heat sink (and current I-), and two temperature (and voltage) probes that are along the length of the sample. The TTO system takes both thermal and electrical measurements by using the same probes, so measurements of thermal conductivity, thermopower, and electrical resistivity can be performed in one pass without remounting the sample.

It is important that the resistance—either thermal or electrical—at the interface between the leads and the sample be minimized. This is especially important when a two-probe measurement (Section 4.3.1) is performed, because any contact resistance is directly reflected in the measured sample thermal and electrical resistance. In addition, you are advised to minimize the thermal diffusion time in the leads by keeping them short (2–3 millimeters, if possible), because this expedites the measurement process.

4.2.1 Geometry

The geometry of the sample is constrained due to a variety of considerations, the most obvious of which is the Thermal Transport sample puck. Mounted vertically on the puck, a sample cannot be much longer than 20 mm, while the minimum convenient sample length is typically 3 mm.

Another aspect to consider is the thermal diffusion time in the sample, defined as $\tau \sim C/K$, where C (Joule/K) is the heat capacity and K (Watt/m) is the thermal *conductance* of the sample. This places an operational lower limit on K so that the measurement time does not become excessively long (one measurement is typically designed to be $\sim 8 \times \tau$; see Section 1.5). Since $K = \kappa \times A/l$, where κ (W/m - K) is the thermal *conductivity*, A is the cross-sectional area of the sample, and l is the length, this implies a lower limit on A/l for a given value of κ . Another relation that can be easily derived from the above equations is $\tau \sim c_p \times l^2/\kappa$, where c_p (J/m³ - K) is the specific heat of the material. This implies that, for a given material, the time constant simply scales quadratically with the length, with a typical practical upper bound of $l_{max} \sim 10$ mm on samples.

While the thermal diffusion time τ places a lower limit on A/l , the heater power $P(W) = \kappa \times \Delta T \times (A/l)$, where $\Delta T \sim 0.03 \times T$ (typical value) is the temperature drop across the sample, sets the upper limit on A/l because the heater is limited by the 10-V compliance limit of the Model 7100 current source. For an $R = 2$ - k Ω chip heater, $P_{max} = V^2/R = 50$ mW.

Keep in mind that the constraints mentioned here are most stringent at high T, where τ is generally longer and the required ΔT is larger. Table 4-1 gives some examples of sample geometries and the range of measurable thermal conductivities based on the above considerations and using a 2-k Ω heater.

Table 4-1. Sample Geometries and Range of Measurable Thermal Conductivities

SAMPLE	DIMENSIONS $L \times (A)$ (mm ³)	HIGH T CONSTRAINTS ON κ (W/m - K)
Brick	8 × (2 × 2)	2–30
Needle	10 × (1 × 1)	10–150
Pill	3 × (5 × 5)	0.1–1.5

4.2.2 Lead-Mounting Epoxies

After deciding on an appropriate geometry for the particular thermal transport measurement you will take (see Section 4.2.1), you cut and sand the sample so that its surfaces are clean.

The epoxies used to attach leads to samples should be chosen for the following:

- Strength in bonding to the particular sample material
- High thermal conductivity
- Convenience of the curing schedule

In addition, any time the thermoelectric or electrical properties will be measured, an electrically conducting silver-filled epoxy must be used. On the other hand, the primary advantage of electrically nonconductive epoxies is their very high strength.



Use a generous (but not excessive) amount of epoxy when attaching leads so that the bond is strong and provides very good thermal contact.

The sample-mounting kit contains starter samples of epoxies, including Silver-Filled H20E from Epoxy Technology, Inc., and nonconductive Tra-Bond 816H01 from Tra-Con, Inc. Product specifications and Materials Safety Data Sheets (MSDS's) for both epoxies are included in the epoxy kit.

4.2.2.1 SILVER-FILLED H20E EPOXY

Small amounts of parts A and B (labeled on the containers) should be thoroughly mixed in an approximately 1:1 ratio on a clean, dry, and nonabsorbing surface, using care that no cross-contamination of the remaining portions in the jars occurs. After applying the mixture to the leads and sample and attaching the leads, you can bake the sample at ~ 150°C for about 5 minutes. See the vendor's product data sheet included in the epoxy kit for more information, including other possible curing schedules.

4.2.2.2 TRA-BOND 816H01 EPOXY

This electrically nonconductive epoxy is provided in one-use, 2-gram packets. Because 2 g is a much larger amount of epoxy than is needed for one sample, Quantum Design recommends preparing several samples at the same time. To mix, slide off the plastic clamp and knead both chambers thoroughly so that the texture feels uniform; this occurs after approximately 2 minutes of mixing. Cut open one end of the packet to dispense epoxy, and allow 24 hours for epoxy bonds to cure. Note that the liquid epoxy may have a grainy appearance; this is normal.

4.3 Two-Probe and Four-Probe Lead Configurations

There are two methods of mounting the electrical/thermal leads on a sample. These two different methods present a trade-off between convenience of mounting and accuracy of measurement.

4.3.1 Two-Probe Lead Configuration

The two-probe lead configuration method is the most convenient because it involves attaching only two leads, but this method sacrifices accuracy because heater/I+ and $T_{hot}/V+$ share one lead while coldfoot/I- and $T_{cold}/V-$ share the other lead (Figure 4-1). Thus, the thermal and electrical contact resistances between the leads and sample contribute to the measured quantities. You should use the two-probe lead configuration method only when the thermal and electrical resistances of the sample are far greater than those of the leads. Examples of samples mounted in this fashion are shown in Figure 4-1, where both bar-shaped and disk-shaped copper leads are used.

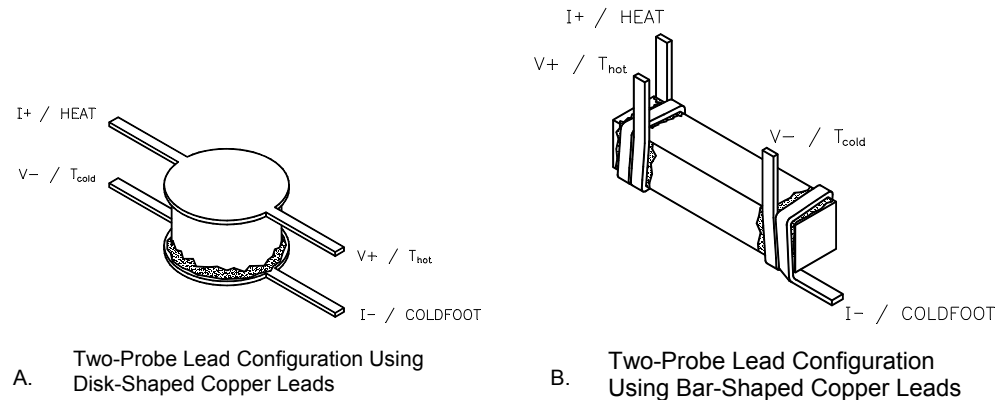


Figure 4-1. Examples of Leads Mounted in Two-Probe Configuration

Note that the thermal conductance of the epoxies decreases very rapidly below 100 K, so the thermal contact resistance may be significant at low temperature even if it is not at room temperature. If you know the cross-sectional area and the approximate thickness of the epoxy used, then you can estimate the contact resistance due to the thermal resistance of the epoxy using the data in Table 4-2 and the equation for the thermal resistance:

$$1/K = 1/\kappa \times l/A,$$

where A is the cross-sectional area of the bond and l is the thickness of the epoxy in the bond.

Table 4-2. Approximate thermal conductance of epoxies

TEMPERATURE (K)	SILVER-FILLED H20E EPOXY κ (W/m - K)	TRA-BOND 816 H01 EPOXY κ (W/m - K)
300	2	1
100	1	0.5
30	0.5	0.2
10	0.2	0.09
5	0.1	0.03
2	0.03	0.01

4.3.2 Four-Probe Lead Configuration

Use a four-probe lead configuration method when sample resistivity (thermal or electrical) is too low to allow you to neglect the contribution of lead contact resistance. Thus, the four probes are attached individually and you avoid the problem of contact resistance at the $T_{hot}/V+$ and $T_{cold}/V-$ probes. This is because very little thermal or electrical current passes into the copper shoes from the sample and hence they approximate much better the ideal of passive probes of the sample's temperature and voltage profile. Note that care must be taken to keep separate the epoxy pads on the sample, or else this advantage is compromised due to thermal/electrical currents that may shunt through the epoxy pads at the T/V probes. Figure 4-2 shows a sample mounted in the four-probe configuration.

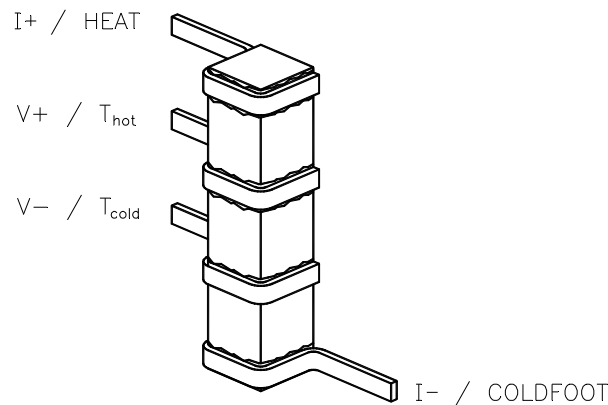


Figure 4-2. Example of Leads Mounted in Four-Probe Configuration

4.4 Checking the Sample Contact

If electrical resistivity or thermopower measurements will be performed on a sample, electrical contact to the sample must be checked after mounting it on the puck. Plug the puck into the puck-wiring test station and check contact at V+/- with an ohmmeter (use the Thermal Transport overlay on the puck box). For resistivity, the contacts at I+/- must also be checked. Finally, check that none of these leads are shorted to the puck body because this may introduce noise into the electrical measurements.

4.5 Using the Puck-Mounting Station

A puck-mounting station (Figure 2-2) is included in the user's kit to make mounting samples on the Thermal Transport puck more convenient. The heavy steel base provides stability while the puck is mounted in the plastic socket, and you can tighten the thumbscrews on the station once you have set the desired orientation of the puck. The plastic socket also acts as a gauge of the copper contact finger positioning on the puck: If the puck is not held in the socket or if it cannot fit in the socket, you must set the finger position with the puck finger adjustment tool (see Section 7.2).

Measurements

5.1 Introduction

This chapter contains the following information:

- Section 5.2 explains how to take measurements with the TTO system.
- Section 5.3 discusses the measurement mode parameters.
- Section 5.4 discusses the TTO measurement process.

5.2 Taking Thermal Transport Measurements



NOTE

Thermal Transport measurements can be taken only if (a) the Thermal Transport connection cable is plugged into the gray, color-coded Lemo port on the PPMS probe head and (b) the Thermal Transport option is activated in PPMS MultiVu. Refer to Appendix A to install and activate the Thermal Transport option.



NOTE

You are encouraged to use the Thermal Transport control center to perform all normal TTO system operations. The automated routines in the control center help ensure that you complete the necessary procedures when you install new samples and create data files. The examples of immediate-mode measurements in this chapter illustrate use of the control center.

5.2.1 Connect Leads to the Sample

Four leads must always be connected to any sample that will be measured with the TTO system: a source (current I+ and heat source), two probes ($T_{\text{hot}}/V+$ and $T_{\text{cold}}/V-$), and a sink (current I- and heat sink). Leads are designed to mount permanently on the sample and offer high thermal conductance and electrical conductivity from the gold-plated copper shoes to the sample.

Complete the following steps to connect leads to the sample:

1. Prepare a sample in which the estimated room-temperature thermal conductance is $K \sim 10 \text{ mW/K}$ (for best results, stay between 5 mW/K and 15 mW/K).
2. Decide whether to attach the leads in a two-probe or four-probe configuration. Refer to Section 4.3 for more information.

For leads, both bars and disks of gold-plated copper are included in the sample-mounting kit. If you use another lead material, it must have high thermal conductance yet be smaller than 0.5 mm (.020 inch) in diameter in order to fit in the shoe assemblies. In addition, it must have good electrical conductivity and be free of insulating oxidized surfaces where contact is made to the shoe assemblies or to the sample.

3. Decide which epoxy material—silver-filled or insulating—is appropriate to the measurements you will take. Refer to Section 4.2.2 for more information.
4. Prepare the leads by bending them to shape so that they fit snugly around the sample to maximize thermal conductance at the contact to the sample.
5. Mount and epoxy the leads and cure them appropriately.

5.2.2 Measure the Sample Dimensions

1. Measure the length l between the hot and cold thermometer probes as well as the cross-sectional area A of the sample in the region between these probes.
2. Calculate the total surface area of the sample and leads as well as an estimate of the sample's infrared emissivity ϵ . This is necessary only for thermal conductivity measurements where some heater power is lost at high T ($> 300 \text{ K}$) to radiative thermal conduction from the hot end of the sample to the surrounding isothermal shield. If the infrared emissivity of the sample is not known, you can often employ these crude approximations:
 - For nonmetallic surfaces—for example, ceramics and heavily oxidized metals— $\epsilon \sim 1$
 - For unpolished metallic surfaces, $\epsilon \sim 0.3$
 - For highly polished metallic surfaces, $\epsilon \sim 0.1$

If thermal radiation from the sample is a concern, you can minimize it by reducing the sample surface area or coating the sample with a thin film of known emissivity, such as varnish—assuming this does not affect other physical properties of interest to you. The infrared emissivities of several substances (for example, some metals, oxides, and paints) are tabulated in the *CRC Handbook of Chemistry and Physics*.



An artifact from thermal radiation can be seen as T^3 “tail” in the thermal conductance that is visible at temperatures above $\sim 200 \text{ K}$. Radiation from the sample and the shoe assemblies is corrected for in the data from the *Conductance* (W/K) and *Conductivity* (W/m - K) columns in the data file. However, no corrections are made in the *Raw Conductance* (W/K) data. Section 1.5.5 explains how to correct for heat losses in TTO measurements.

5.2.3 Mount the Sample

1. Place the puck in the Thermal Transport puck-mounting station and clamp the I-/coldfoot sample lead to the coldfoot on the puck by using the small Phillips screwdriver included in the user's kit.
2. Affix the shoes to the three remaining leads consistent with Figure 4-1 or 4-2 by using the small slotted screwdriver and tweezers to hold the probe shoes. Use enough force to make the M1 screws snug, but do not overtighten them because overtightening damages the soft copper of the probe shoe. Note that the white connector plug on the $T_{\text{hot}}/V+$ shoe assembly plugs into the puck at the middle socket which is painted red, while $T_{\text{cold}}/V-$ plugs into the blue socket. As a convenience in sample mounting, you may use the red and blue Sharpie permanent markers included with TTO to color the white surfaces on both ends of the hot and cold thermometer shoe assemblies.
- * Note: When attaching the heater shoe, make sure the lead does not touch the heater resistor's solder pad, potentially causing an electrical short.
3. Note the serial numbers of each of the three shoe assemblies. These numbers are handwritten on one side of the connector plug at the puck.

**NOTE**

While attaching the shoes, use care not to pull on the thin wires running to the probe shoes, and avoid scratching the areas near solder pads, especially where the wire is soldered to the back wall of a shoe.

4. Check carefully that neither the shoes nor their wires are touching each other or any part of the puck, and that the sample is contacting the puck only at the clamp of the coldfoot.
5. Place the radiation shield on the puck, remove the shield cap, and inspect to verify that no wires, shoes, or that the sample touches the shield.
6. Replace the shield cap before you insert the sample into the PPMS probe.

5.2.4 Install the Sample

1. Activate the Thermal Transport option if the option is not currently active. Do the following: (a) select **Utilities**►**Activate Option** in the PPMS MultiVu interface, (b) click on **Thermal Transport** under the **Available Options** heading, and then (c) select **Activate**.
2. Select the **Install** tab in the Thermal Transport control center (Figure 3-2).
3. Select **Install Wizard** and follow the software prompts to install the sample in the PPMS sample chamber.

5.2.5 Start the High-Vacuum System

If the install wizard did not start the high-vacuum system, you start the high-vacuum system by doing one of the following: either (a) select the **HiVac** button in the **Install** tab in the Thermal Transport control center or (b) select **Instrument**►**Chamber**►**HiVac** in the main PPMS MultiVu interface. Wait for the HiVac state to be reached before starting thermal transport measurements, because a significant heat leak can result from gas thermal conduction away from the TTO heater.

5.2.6 Open the Data File

1. Select the **Data File** tab in the Thermal Transport control center (Figure 3-3). The tab indicates which data files are selected to save the measurement data. If you run the measurement when no data file is selected, the data is discarded.
2. Click **Browse** to select a different file or create a new file. **Browse** opens the **Thermal Transport Select Data File** dialog box, which lists all existing files.
3. Select a data file or enter the name of a new file. When you create a file, the software prompts you to define the sample properties for the sample whose measurement data will be saved to the file, and the data entry fields in the control center's **Sample** tab are enabled. Define the sample properties, and then select **OK**.

As soon as you select or create a file, the **Data File** tab appears again, and the **File Name** field identifies the data file you have selected. Notice that by default the **Capture Raw Data** check box is *not* selected. If this box is enabled, raw measurement data is saved to a raw data file that has the same name as the measurement data file but a `.raw` file extension instead of a `.dat` file extension.

5.2.7 Define the Measurement

1. Decide which mode—continuous or single—to use for the measurement. Continuous mode is the software default and generally offers the most rapid data acquisition. Single mode offers the most direct control of the measurement. To specify single mode measurements, use the **Mode** tab of the **Thermal Transport Measurement** dialog (Figure 3-10). Single mode measurements may be steady state or timed. Refer to Section 5.4 for more details about each measurement.
2. Decide whether to take the measurements directly by accessing the **Thermal Transport Measurement** dialog (select the **Measure...** button in the control center) or to run the measurements in a PPMS MultiVu sequence.
3. Select the **Measure...** button in the control center so that you see the **Settings** tab in the **Thermal Transport Measurement** dialog (Figure 3-7). You use the **Settings** tab to decide which physical quantities you want to measure: thermal conductivity, Seebeck coefficient, electrical resistivity, and (if all the above are selected) whether you would like the program to compute the figure of merit ZT from these measurements and put this quantity in the data file.
4. Define appropriate parameters for each measurement. The default values for these parameters are appropriate for most cases, so it is unlikely you will need to change them.
 - In continuous mode, which uses an adaptive algorithm, you must set the allowable ranges for thermal measurement parameters. These parameters, which include measurement period, heater power, and Seebeck voltage, are set in the **Thermal** tab in the **Thermal Transport Measurement** dialog (Figure 5-1). Similarly, for resistivity measurements you must define the range of excitation and frequency parameters. You set these parameters in the **Resistivity** tab in the **Thermal Transport Measurement** dialog (Figure 5-2).
 - In single mode, you state the style of single mode measurement along with the fixed heater power, measurement period, and maximum expected Seebeck voltage.

5.2.8 Run the Measurement

5.2.8.1 RUNNING THE MEASUREMENT INTERACTIVELY

To run a thermal transport measurement interactively, you select the **Measure...** button that is at the bottom of the Thermal Transport control center. Selecting the **Measure...** button opens the **Thermal Transport Measurement** dialog box, which you use to select the thermal properties you want to measure as well as the parameters and limits for the measurements. By default, dynamic (continuous) measurements are made using an AC technique unless you use the **Mode** tab (Figure 3-10) to switch to steady-state (single) measurements. Refer to Section 1.3 for more information on the various modes of operation. The measurement does not run until you select the **Start** button in the **Thermal Transport Measurement** dialog.

Data from any measurement is automatically saved if a data file is open.

5.2.8.2 RUNNING THE MEASUREMENT IN A SEQUENCE

To run a measurement in sequence mode, you run a sequence that contains **TTO Measure** commands (under **Measurement Commands**). The measurement is taken automatically when PPMS MultiVu reads the measurement command in the running sequence.

Thermal measurements in sequence mode can generally be taken in two ways:

- Slowly ramp the temperature (rate ~ 0.5 K/min.) and measure continuously. This method uses the **Set Temperature** sequence command and is usually the more expedient technique.
- Scan in temperature (or magnetic field) and take single measurements after the system has stabilized at the new temperature or field. This method uses the **Scan Temperature/Scan Field** commands.

Data from any measurement is automatically saved if a data file is open.

The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses sequence files and all standard system sequence commands in detail.

5.2.9 Scanning or Ramping the Temperature While Measuring

In either interactive or sequence mode, the most common measurement technique is to start continuous measuring and then ramp the temperature slowly by using the **Set Temperature** command with a slow rate (slew rates are typically ~ 0.1 – 1 K/min.). For example, if the system is at room temperature, you can set a target temperature of 1.8 K and a slew rate of 0.5 K/min.

If you are taking single steady-state measurements, these are obviously taken at a fixed temperature and field. This method is more amenable to the **Scan Temperature** command, which allows you to wait for system stability at each target before taking a measurement.

5.3 Measurement Mode Parameters

5.3.1 Continuous Measurement Mode

Parameters for continuous mode measurements are defined in the **Thermal** tab in the **Thermal Transport Measurement** dialog box. You open this dialog box by selecting the **Measure...** button in the Thermal Transport control center.

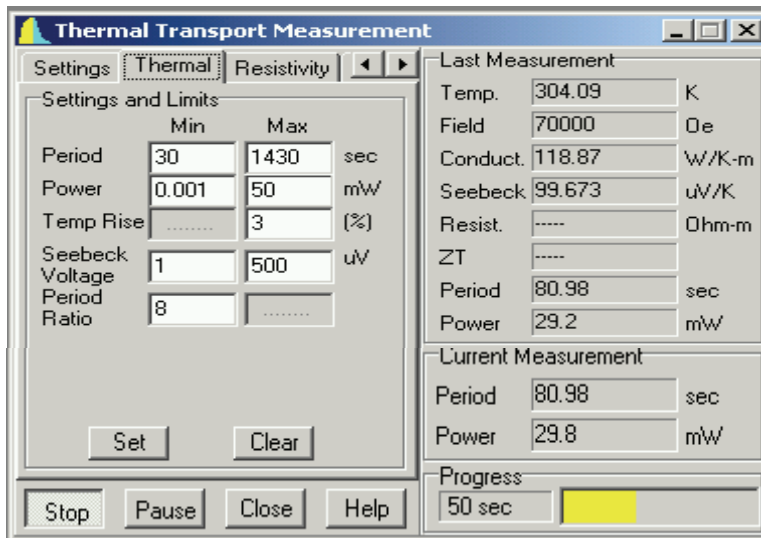


Figure 5-1. Thermal Tab in Thermal Transport Measurement Dialog Box

The **Clear** button in the **Thermal** tab restores either the values that were in memory after the last time the **Set** button in the tab was selected, or the default values if **Set** has not yet been selected.

Table 5-1. Minimum and Maximum Parameter Limits for Continuous Mode Measurements

PARAMETER	FUNCTION	DESCRIPTION
Period	Length, in seconds, of heater on/off cycle.	Minimum is 30 seconds due to limited data acquisition rate of hot/cold Cernox temperatures from user bridge. Maximum period is 1430 seconds. Only discrete values of the period are allowed: period = $4292/n$, where n is an integer ($n > 2$). This implies that jumps between available periods become larger as period grows.
Power	Limits power that heater resistor can output. Expressed in mW.	Default limits are 1 μ W and 50 mW. Minimum value is limited by hardware limitations of the DAC of the current source to 10 μ A, which translates to 0.2 μ W for 2-k Ω heater. Maximum value is set by voltage compliance of 10 V of the current source, which translates to 50 mW for 2-k Ω heater.

(continues)

Table 5-1. Minimum and Maximum Parameter Limits for Continuous Mode Measurements (Continued)

PARAMETER	FUNCTION	DESCRIPTION
Temp. Rise	Target value of temperature rise during heating, expressed in percent of absolute temperature. Heater power is adaptively adjusted to achieve the desired Temp. Rise.	Defined as rise in T_{hot} due to the heater, divided by average temperature during this time. This parameter can be decreased in order to minimize uncertainty in temperature during a pulse, such as in cases where physical properties are changing very rapidly with temperature. Errors in data increase sharply if Temp. Rise < 1% is chosen, due to small magnitude of thermal signal.
Seebeck Voltage	Determines initial gain settings, in μV , for DSP voltage readback preamp.	Entering an expected maximum value determines initial gain settings for DSP voltage readback preamp. Software uses “sticky autorange” algorithm to rescale preamp if initial guess was far off.
Period Ratio	Provides feedback for heater period.	Defined as ratio of the period to long time-constant τ_1 of sample. Default period ratio value of 8 has been found empirically to be near minimum needed by curve-fitting algorithm in order to converge on correct result.

Table 5-2. General Settings for Continuous Mode Measurements

PARAMETER	FUNCTION
Save Marginal Results	Prompts software to write to data file results for which fitting algorithm was able to converge on a value but encountered significant errors from one or more sources. Results are deemed marginal if the error on the curve fit is between 50% and 200%.
Discard First N Results	Prompts software to discard user-specified number of first results. This setting is useful because first several (~ 3) data points are usually of poor quality because parameters were still in the process of being optimized.

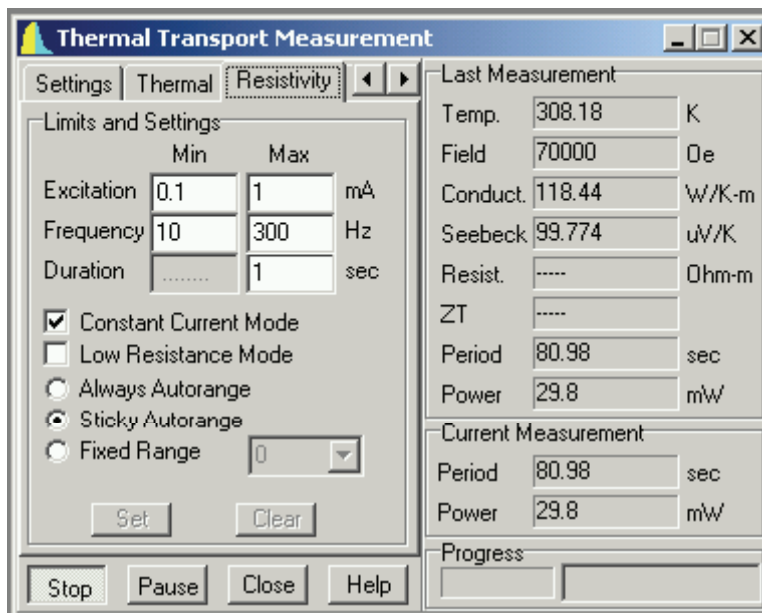


Figure 5-2. Resistivity Tab in Thermal Transport Measurement Dialog Box

Table 5-3. Resistivity Excitation Parameters for Continuous Mode Measurements

PARAMETER	FUNCTION
Excitation	Defines minimum and maximum excitation amplitude in milliamps.
Frequency	Defines minimum and maximum measurement frequency in Hz.
Duration	Defines measurement duration in seconds.



NOTE

The parameters for resistivity measurements are adjusted adaptively in the continuous measurement mode, in the same way as the heater power and period are allowed to vary. Refer to the *Physical Property Measurement System: AC Transport Option User's Manual* for resistivity settings.

5.3.2 Single Measurement Mode

The parameters for single mode are generally simpler than the parameters for continuous mode. Basically, you fix the heater power and set a heater on/off criterion that depends on the style of measurement. In **stability** mode, the criterion is the temperature stability on both the hot and cold thermometer probes. In **timed** mode, you set a period length for the heater pulse that can be as long as desired. Finally, for **Seebeck voltage** you enter an estimate of the change in thermal voltage (in μV) that you expect from the sample during the measurement. This determines the gain settings on the DSP circuitry in the AC Transport controller.

5.4 Description of Measurement Process

With the TTO system you can measure thermal properties in either continuous mode or single mode. The conceptual distinction between the two is that in continuous mode, the system is constantly measuring the thermal properties and any acceptable data is saved to the data file, while in single mode the system takes a measurement only when you request that it do so. An advantage of the continuous technique is that a higher density of data is obtained, and you take advantage of the adaptive algorithms built into the software which are constantly optimizing the system parameters for each measurement. However, it may be preferable in some cases to have more direct control of the measurement—for example when one measurement takes several minutes or when the adaptive software routines may not adequately adjust to rapidly changing thermal transport properties of the sample—and in these cases you want to use single mode.

In continuous measurement mode, the parameters of heater power and period as well as the resistivity excitation and frequency are continually being updated after each heater pulse to keep the Temp. Rise and Period Ratio parameters near the user-set values. The raw data $\Delta T(\text{time})$ is sent to the fitting algorithm, which performs a three-parameter, nonlinear, least-squares fit in ΔT_∞ , the asymptotic temperature drop across the sample, as well as τ_1 and τ_2 , which are a long and short time constant, respectively, that characterize the sample-lead-shoe thermal circuit. Of these, ΔT_∞ is used to calculate the thermal conductance of the sample $K = (\text{heater power})/\Delta T_\infty$, while the period for the next measurement is computed from τ_1 by $\text{Period} = \text{PeriodRatio} \times \tau_1$. The asymptotic Seebeck voltage ΔV_∞ is computed from the raw data $\Delta V(\text{time})$ similarly, except that a computationally simpler linear regression is used because τ_1 and τ_2 are based on the conductivity fitter routine. The Seebeck coefficient is then simply $\alpha = \Delta V_\infty/\Delta T_\infty$. Resistivity measurements are made before and after the heater pulse, and the average of the two is taken as the sample resistivity. If the thermoelectric figure of merit is sought, then $ZT = \alpha^2 \times T/(\kappa \times \rho)$ is computed and written to the data file.

Single measurements, which are generally most useful to advanced users with specialized requirements, are subdivided into stability and timed measurement methods. In the stability technique, you specify requirements for thermal stability at the hot and cold thermometer probes on the sample and a user-defined heater power is applied only after the stability criterion is met. Temperature and/or voltage data points are taken in the quiescent state before the heater is turned on, and heater power is applied until thermal stability is once again achieved at both thermometer probes, at which point the second series of data points is taken. In a timed measurement, you set a fixed period and power for one heater pulse. Note that in all these single-mode thermal measurements, data points are taken only at the end of the settling time and at the end of the heater “on” segment, and it is assumed that $\Delta T = \Delta T_{\text{on}} - \Delta T_{\text{off}}$ and $\Delta V = \Delta V_{\text{on}} - \Delta V_{\text{off}}$ represent asymptotic steady-state values; that is, no curve fitting of the data is performed.

See Section 1.5 for more information on the theory of operation for the Thermal Transport option.

Troubleshooting

6.1 Introduction

This chapter contains the following information:

- Section 6.2 offers suggestions to help troubleshoot jumps or noise in TTO data.
- Section 6.3 discusses ways to minimize errors due to radiation effects.
- Section 6.4 refers to high-vacuum problems.

6.2 Jumps or Noise in the Data

The following are some general guidelines for ensuring good data quality:

1. Make sure that the condition Period Ratio ($= \text{Period}/\tau_1$) > 8 is met at all temperatures. Considering that the maximum period is 1430 seconds, your sample must be designed so that the thermal diffusion time τ_1 is not too long. If Period Ratio is too small, the curve-fitting software is not able to adequately fit the data.
2. Make sure that an adequate heat pulse can be applied (default is Temp. Rise = 3%) across the sample; that is, the thermal conductance of the sample is not above about 20 mW/K. If Temp. Rise falls below the 1% level, data can become noisy.
3. Verify that the leads are attached to the sample with a generous amount of epoxy that is well cured and that the lengths of the leads are kept to a minimum.
4. If temperature or field is being slewed while measurements are made, verify that the slew rate is slow (typically less than 1 K/min.) and uniform over the course of a measurement.

6.2.1 Gaps in the Data Versus Temperature

- Check that the background temperature slewing of the system as reflected in the hot and cold sample thermometers and the system thermometer (see the `.raw` data file) was linear and not irregular.
- Check the value for the period of thermal measurements in the vicinity of the gap, because a very long period results in lower density of data.
- Look in the TTO **Error Count** and TTO **Status Log** dialogs for evidence of errors. Also check the codes for both the PPMS (`Status`) and TTO (`Error`) status in the data file and consult Appendix B to interpret the codes.

6.2.2 Steps in the Data

If the data jumps in a step-wise fashion as a function of temperature or field, do the following:

- Check to see if the jump exists only in the magnitudes of thermal conductance and electrical resistance, but is absent in Seebeck coefficient data. This is evidence that the sample geometry (A/l) has changed due to internal cracking of the sample (this can often occur under temperature cycling) or breaking of the epoxy bonds to the leads. Weak epoxy bonds are evidence of poor epoxy strength or poor thermal matching of the epoxy and the sample.
- Look at the standard deviation in the measured quantities and also at the `.raw` data to see if the software is adequately modeling the data. A step in the data can occur when the data fitting is poor enough that several distinct solutions have comparable curve fit errors. See the `ERROR` column in the `.dat` file and consult Appendix B to interpret the TTO status code.

6.3 Thermal Radiation “Tail” in the Thermal Conductivity Data

Thermal radiation between the sample (and shoe assemblies) and the surrounding environment introduces errors in the measurement of thermal conductivity at high temperatures. Some of the heat produced by the heater resistor radiates instead of traveling through the sample, in accordance with the radiation law described in Section 1.5.5. The resulting “tail” has a T^3 temperature dependence that is generally observable only above about 200 K. Described here are some ways of minimizing errors due to radiation effects. You may try one or more of these techniques in order to manage thermal radiation in your measurements:

1. Increase the geometrical factor of the sample A/l so as to make the thermal conductance of the sample much higher than the errors associated with subtracting the radiation thermal conductance. These errors are about ± 1 mW/K at the highest temperatures.
2. Minimize errors in estimating sample radiation by coating the surface of the sample with a material of known emissivity, or by choosing a sample geometry minimizing the radiation surface area.
3. Modify the mounting of leads on the sample by thermally sinking the heater and hot thermometer shoes together, and connect only the heater to a lead on the sample. The shoes can be affixed to each other by using H Grease to stick them together back-to-back, and by making sure that they do not tend to separate. When the shoes are placed in this special configuration, it is recommended that you take measurements in single mode because the fitter algorithm used in continuous mode often does not adequately model this thermal circuit. The reason this technique works is that the stray conductance due to the shoe assemblies, K_{shoes} , was estimated in a configuration in which the heater and hot thermometer were isothermal. However, in real samples the heater will always be hotter than the hot thermometer so that some thermal radiation from the heater is not entirely accounted for.

6.4 High-Vacuum Problems

For help troubleshooting the PPMS High-Vacuum option, please refer to the appropriate manual, either the *Physical Property Measurement System: Turbo Pump High-Vacuum Option User's Manual* or the *Physical Property Measurement System: Cryopump High-Vacuum Option User's Manual*, depending on the type of high-vacuum system that you have.

Maintenance

7.1 Introduction

This chapter contains the following information:

- Section 7.2 explains how to use the puck adjustment tool.
- Section 7.3 explains how to grease the puck fingers and the coldfoot clamp.

7.2 Using the Puck Adjustment Tool

The puck adjustment tool (Figure 7-1) adjusts the tension in the chuck fingers so that the fingers maintain solid thermal contact with the heater block located at the bottom of the sample chamber. Solid thermal contact between the chuck fingers and the heater block is especially important for high-vacuum applications, such as heat capacity and thermal transport measurements.

The puck adjustment tool consists of two metal cylinders. In Figure 7-1, cylinder 1 is the finger spreader, and cylinder 2 is the finger contractor and the test cutout. The finger spreader and the finger contractor adjust the tension of the chuck fingers. The test cutout, which has the same dimensions as the cutout in the heater block, tests how well the chuck fingers will contact the heater block.

You use the puck adjustment tool on the puck after you have inserted the puck into the sample chamber approximately 10 times or whenever the puck fits loosely into the bottom of the sample chamber.

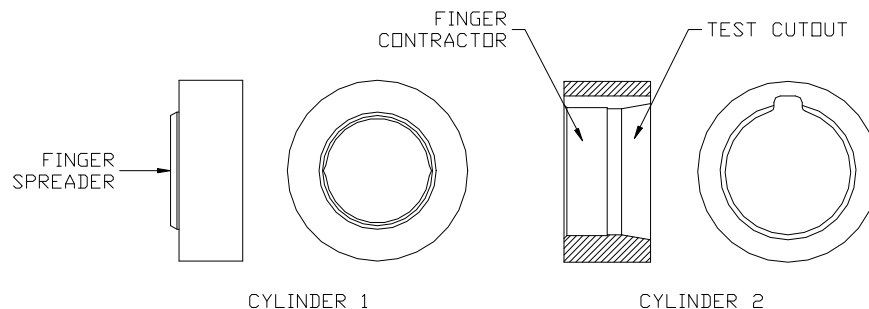


Figure 7-1. Puck Adjustment Tool

Complete the following steps to use the puck adjustment tool:

1. Screw the thermal radiation shield onto the TTO puck.
2. Place the puck on the finger spreader. Refer to Figure 7-1. Turn the puck until the screw heads on the bottom of the puck line up with the grooves inside the finger spreader. Press the puck downward and continue pressing until all chuck fingers touch the base of the finger spreader. When all fingers touch the base of the spreader, the spreader evenly applies radial force to the fingers, pushing them outward and slightly beyond their optimal location.
3. Remove the puck from the finger spreader.
4. Place the puck inside the finger contractor. Refer to Figure 7-1. Press straight down on the puck and continue pressing until you press the puck completely into the finger contractor. When the entire chuck is in the contractor, the contractor evenly applies force to the outside of the fingers, pushing them inward. The contractor pushes the fingers—regardless of external wear or variations on the puck—so that the fingers obtain their optimal location.
5. Remove the puck from the finger contractor.
6. Place the puck inside the test cutout. Refer to Figure 7-1. Verify that the puck fits easily but snugly in the test cutout.

7.3 Greasing the Puck Fingers and the Coldfoot Clamp

The thermal contact between the sample puck and the heater block can be further improved by applying a small amount of H Grease (which is included in the TTO user's kit) to the puck fingers. You may also wish to use a small amount of H Grease on the sample's cold lead when clamping it in the coldfoot, because this contact can be a significant thermal resistance. Note that it is always important to apply enough tension to the coldfoot screw in order to have good thermal and electrical contact between the sample lead and the coldfoot.

Installation

A.1 Introduction

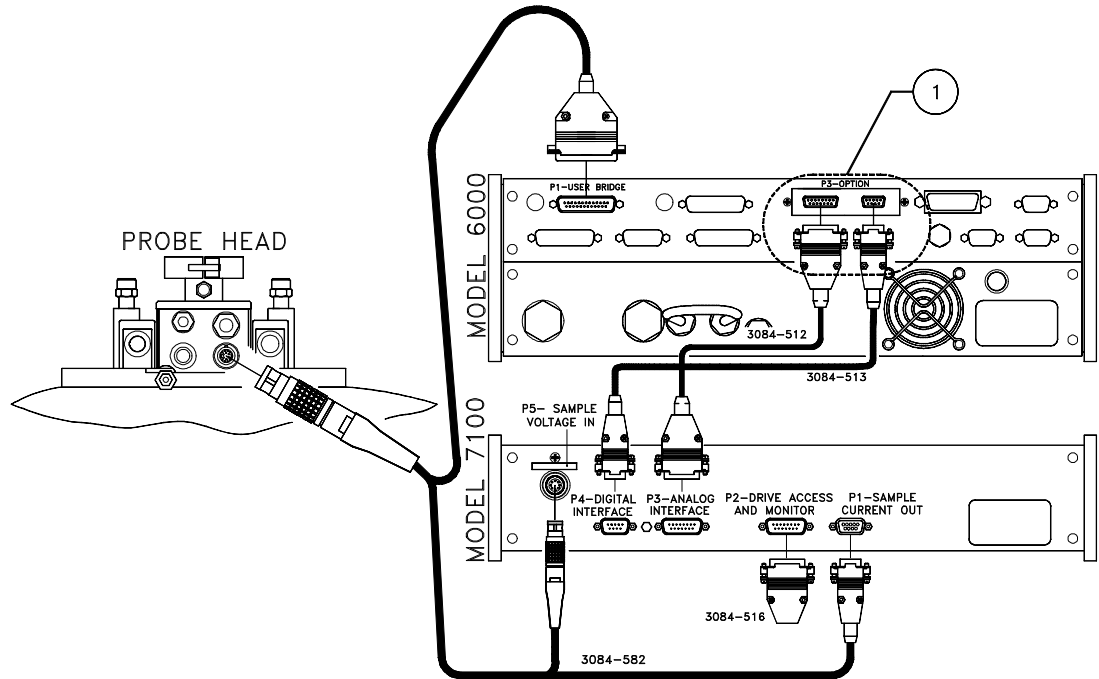
This appendix contains the following information:

- Section A.2 explains how to install the TTO hardware.
- Section A.3 explains how to install the TTO software.

A.2 Installing Thermal Transport Hardware

To install the TTO hardware, you must install all components that are necessary for the TTO system. These components may include the following:

- **Model 7100 AC Transport Controller**
For more information about the Model 7100, refer to section 2.3.1 in this manual and to the *Physical Property Measurement System: AC Transport Option User's Manual*.
- **High-Vacuum option**
The TTO system works with either the Turbo Pump High-Vacuum option or the Cryopump High-Vacuum option. For more information about these options, refer to the *Physical Property Measurement System: Turbo Pump High-Vacuum Option User's Manual* or the *Physical Property Measurement System: Cryopump High-Vacuum Option User's Manual*.
- **User bridge board**
For more information about the user bridge board, refer to section 2.2.6 in this manual and to the *Physical Property Measurement System: Resistivity Option User's Manual*.
- **Thermal Transport connection cable**
This cable must be connected as described in section 2.2.5 and as illustrated in Figure A-1 on the following page.
- **WaveROM EPROM (certain systems only)**
If the ACMS waveROM upgrade kit was included with your TTO system, you must install the new ROM. Refer to sections 2.2.4 and 2.2.4.1.



① THESE CABLES MAY GO TO THE OPTION CONTROLLER
DEPENDENT ON SYSTEM CONFIGURATION.

Figure A-1. Thermal Transport Option Connection Diagram

A.3 Installing Thermal Transport Software

1. Install the PPMS MultiVu software (version 1.1.6 or later) if it is not already installed. Do the following: (a) insert PPMS MultiVu Disk 1 into the PC, (b) select the A: drive, (c) select `setup.exe`, and then (d) complete all operations the InstallShield wizard prompts you to perform.

The TTO software runs in conjunction with the PPMS MultiVu software. PPMS MultiVu must be installed on the host computer in order for the TTO software to work. If you try to install the TTO software before you install PPMS MultiVu, the InstallShield wizard for the TTO software fails and generates a warning message, which tells you to install PPMS MultiVu.

2. Install the TTO software. Do the following: (a) insert Disk 1 for the TTO software into the PC, (b) select the A: drive, (c) select `setup.exe`, and then (d) complete all operations the InstallShield wizard prompts you to perform.
3. Activate the Thermal Transport option in PPMS MultiVu. Do the following: (a) start up PPMS MultiVu, (b) select **Utilities**►**Activate Option**, (c) click on **Thermal Transport** under the **Available Options** heading, and then (d) select the **Activate** button.

As soon as you activate the Thermal Transport option, the Thermal Transport control center opens and the **Measure** menu items and measurement sequence commands that are specific to TTO appear in the PPMS MultiVu interface.



Note that in order to run, TTO requires that the AC Transport (ACT) option be installed because TTO and ACT share the hardware configuration file

C:\QdPpms\ACTrans\Calibration\Actcal.cfg. For information about installing the ACT option, refer to the *Physical Property Measurement System: AC Transport Option User's Manual*.

Status Codes and Error Messages

B.1 Introduction

This appendix contains the following information:

- Section B.2 lists the system status codes for the PPMS and for TTO.

B.2 System Status Codes

B.2.1 General PPMS System Status Codes

Table B-1. Status Associated with Bits of General System Status Field:
Description of General System Status Measure Codes

BITS	VALUE	TEMPERATURE STATUS
0-3	0	Status unknown.
	1	Normal stability at target temperature.
	2	Stable.
	5	Within tolerance; waiting for equilibrium.
	6	Temperature not in tolerance, not valid.
	7	Filling/emptying reservoir.
	10	Standby mode invoked.
	13	Temperature control disabled.
	14	Request cannot complete, impedance not functioning.
	15	General failure in temperature system; contact Quantum Design.

(continues)

Table B-1. Status Associated with Bits of General System Status Field:
Description of General System Status Measure Codes
(Continued)

BITS	VALUE	MAGNET STATUS
4-7	0	Status unknown.
	1	Persistent mode, stable.
	2	Persist switch warming.
	3	Persist switch cooling.
	4	Driven mode, stable at final field.
	5	Driven mode, final approach.
	6	Charging magnet at specified voltage.
	7	Discharging magnet.
	8	Magnet reset.
	9	Current error, incorrect current in magnet.
	10	Persistent switch heater error.
	11	Magnet quench.
	12	Magnet charging error.
	14	Power supply error.
15	General failure in magnet control system.	

BITS	VALUE	CHAMBER STATUS
8-11	0	Status unknown.
	1	Purged and sealed.
	2	Vented and sealed.
	3	Sealed, condition unknown.
	4	Performing purge/seal routine.
	5	Performing vent/seal sequence.
	6	Pre-pump (turbo pump) / High vacuum evacuate (cryopump).
	7	High vacuum.
	8	Pumping continuously.
	9	Pre-vent / Flooding continuously.
	14	High vacuum error.
	15	General failure in gas control system.

(continues)

Table B-1. Status Associated with Bits of General System Status Field:
Description of General System Status Measure Codes
(Continued)

BITS	VALUE	SAMPLE POSITION STATUS
12-15	0	Status unknown.
	1	Sample stopped at target value.
	5	Sample moving toward set point.
	8	Sample hit limit switch.
	9	Sample hit index switch.
	15	General failure.

B.2.2 Thermal Transport System Status Codes

Table B-2. TTO Status Codes

ERROR	BIT FIELDS
Computation : poor curve fit	1-5
Computation : no curve fit	6-10
Reserved	11-16
Fatal Error : software	17-21
Fatal Error : hardware	22-26
Reserved	27-32

In Table B-2, “poor curve fit” means that the residual of the fit was greater than 50% or that the Period Ratio was less than 4, and “no curve fit” means that the software was unable to fit the data to the model.

The five bits for each error category represent, respectively, errors in thermal conductivity, Seebeck coefficient, first resistivity, second resistivity, or figure of merit. At a glance, you can interpret the TTO status code as the following:

code = 0	No errors in measurements or computations
1 < code < 32	One or more poor computed curve fits
33 < code < 1024	One or more failed curve fits
code > 1024	Fatal error

Pinout Tables

C.1 Introduction

This appendix contains the following information:

- Section C.2 discusses and illustrates the pinouts for the TTO system.

C.2 Thermal Transport Pinouts

The following table and diagram detail the pinouts for each connector in the TTO system. The diagram illustrates hardware ports, not connectors at the end of the cables.

C.2.1 Sample Connections

The Thermal Transport connection cable (Figure 2-5) has connections to both the Model 7100 AC Transport controller and the Model 6000 user bridge board. As in the case for the AC Transport (ACT) option, connections from the Model 7100 to the sample are configured to minimize “cross-talk” between the excitation signal (the P1 port on the Model 7100) and the detected signal (the P5 port on the Model 7100). In addition, each “+/-” pair is individually twisted in the gray Lemo cable and also in the wiring between the puck base and the sample. The exceptions to this are the I+/- leads and the V+/- leads between the five-pin connector plugs and the sample (recall that V+ and V- run to separate shoe assemblies, so they are physically separated at this point).

Table C-1 on the following page lists all TTO sample connections. Figure C-1 illustrates the sample connections, and Figure C-2 illustrates the pinout of the connector sockets on the Thermal Transport sample puck.

Table C-1. TTO Sample Connections

FUNCTION	SYMBOL	SAMPLE PUCK / GREY LEMO	P1 PORT ON MODEL 7100	P5 PORT ON MODEL 7100	P1 USER PORT ON MODEL 6000
Current + (thermometers)	I_{Th}^+	3			5 + 7
Current - (thermometers)	I_{Th}^-	4			18 + 20
Current + (heater)	Q+	5	1		
Current - (heater)	Q-	6	6		
Voltage + (cold thermometer)	V_{Th}^{C+}	7			8
Voltage - (cold thermometer)	V_{Th}^{C-}	8			21
Voltage + (warm thermometer)	V_{Th}^{W+}	9			6
Voltage - (warm thermometer)	V_{Th}^{W-}	10			19
Current + (sample)	I+	11	2		
Current - (sample)	I-	12	7		
Voltage + (sample)	V+	13		1 + 3 + 5 + 7	
Voltage - (sample)	V-	14		2 + 4 + 6 + 8	
Ground			5	shell	13

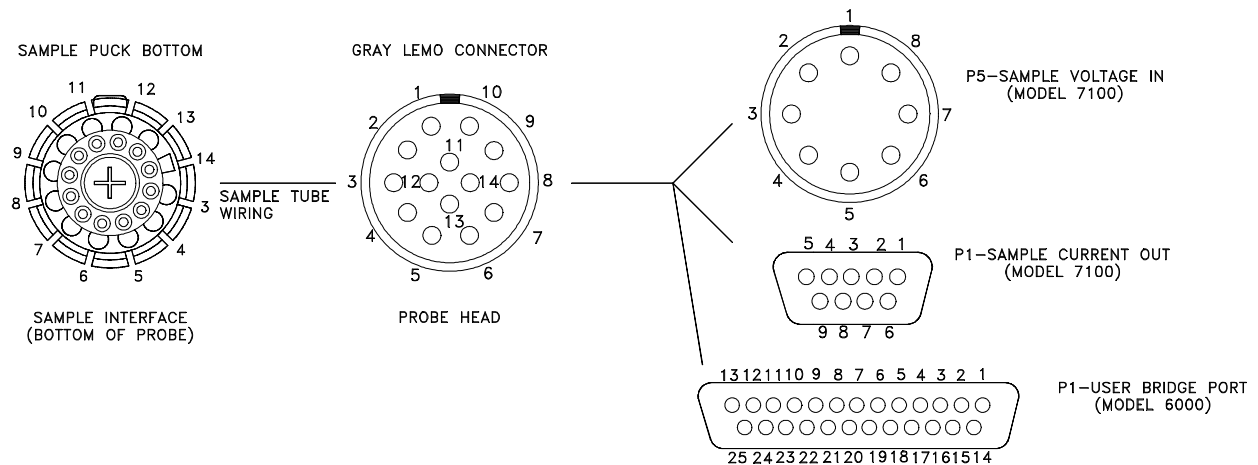


Figure C-1. Illustration of TTO Sample Connections, Showing Hardware Ports

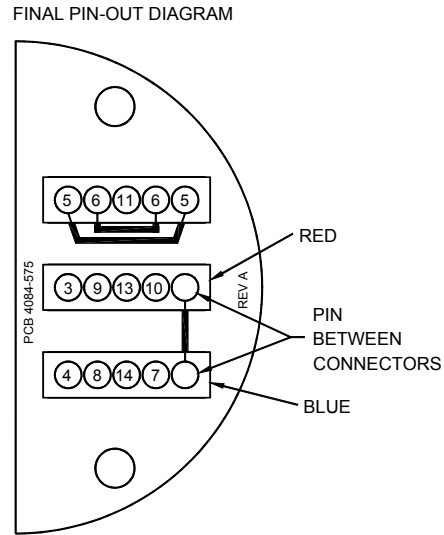


Figure C-2. Top View of Pinout of Connector Sockets on Thermal Transport Sample Puck

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Physical Property Measurement System

Horizontal Rotator Option User's Manual

Part Number 1384-100 B1

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U.S. Patents

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)
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.

P.2 Scope of the Manual

This manual contains important information regarding the care, handling, and use of the Horizontal Rotator option for the Physical Property Measurement System (PPMS).

P.3 Contents of the Manual

- Chapter 1 presents an overview of the Horizontal Rotator option and discusses the Horizontal Rotator probe.
- Chapter 2 explains how to install the Horizontal Rotator hardware.
- Chapter 3 discusses the operation of the rotator motor and how the rotator affects measurements.
- Appendix A lists the PPMS firmware commands that may be used with the Horizontal Rotator.
- Appendix B contains the interconnection tables.

P.4 Conventions in the Manual

File menu	Bold text distinguishes the names of menus, options, buttons, and panels appearing on the PC monitor or on the Model 6000 PPMS Controller LCD screen.
File>>Open	The >> symbol indicates that you select multiple, nested software options.
STATUS	Bold text and all CAPITAL letters distinguish the names of keys located on the front panel of the Model 6000 PPMS Controller.
<code>.dat</code>	The <i>Courier</i> font distinguishes characters you enter from the PC keyboard or from the Model 6000 PPMS Controller front panel. The <i>Courier</i> font also distinguishes code and the names of files and directories.
<Enter>	Angle brackets <> distinguish the names of keys located on the PC keyboard.
<Alt+Enter>	A plus sign + connecting the names of two or more keys distinguishes keys you press simultaneously.
Note	Text is set in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances.

CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.



WARNING!

Text is set off in this manner to signal conditions that could result in bodily harm, loss of life, or irreparable damage to equipment.



WARNING!

Text is set off in this manner to signal electrical hazards that could result in bodily harm or loss of life.

Introduction

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the PPMS Horizontal Rotator option.
- Section 1.3 discusses operation of the Horizontal Rotator probe.

1.2 Overview of the Horizontal Rotator Option

The Quantum Design Horizontal Rotator option for the Physical Property Measurement System (PPMS) allows sample rotations around an axis that is perpendicular to the magnetic field of a longitudinal PPMS magnet. The Horizontal Rotator probe fits into the PPMS sample chamber with all wiring connections accomplished via the keyed connector at the bottom of the sample chamber. The rotator may safely be exposed to all temperatures accessible in the PPMS.

The range of rotator rotation is -10° to 370° . For Horizontal Rotators with serial numbers 011 or greater, 0° and 360° denote the face-up orientation. For Horizontal Rotators with serial numbers 001 to 010, 0° and 360° denote the face-down orientation, and 180° denotes the face-up orientation. An anti-backlash spring helps keep the same gear faces in contact at all times. For the standard rotator motor, the motorized steps are in $.0532^\circ$ increments, and the maximum rate of rotation is 10° per second. For the high-resolution motor, the motorized steps are in 0.00449° increments, and the maximum rate of rotation is about 50° per minute. Jewel bearings on the moving parts lengthen the life span of the rotator.

Table 1-1. Hardware for Horizontal Rotator Option

HARDWARE	PART NUMBER	ILLUSTRATION
Horizontal Rotator Probe	4084-304	Figure 1-2
Resistance Bridge Sample Holder Board	3084-371	Figure 2-1
Support Plate with Motor	4084-302	Figure 2-6
Rotator Support Tool	4084-391	Figure 2-2
Sample Removal Tool	4084-307	Figure 2-3
Horizontal Rotator Kit for ACT Option	4084-308	

The Horizontal Rotator option may be used with the PPMS AC Transport Measurement System (ACT) option or the PPMS Resistivity option. For detailed information about using the rotator with either of these options, refer to the *Physical Property Measurement System: AC Transport Option User's Manual* or the *Physical Property Measurement System: Resistivity Option User's Manual*.

1.2.1 Manual and Automatic Operation

The Horizontal Rotator may be operated manually or used with an automatic motor drive. The vernier dial on the top of the Horizontal Rotator probe may be turned manually, or the motor connected to the support plate can mate with the top of the rotator to control sample orientation. The **Instrument>> Motion** menu option in PPMS MultiVu allows immediate access to motor control and configuration. Sequence commands can automate motor control through the use of PPMS sequences. Section 3.2.2 discusses immediate-mode operation. Section 3.2.3 discusses sequence mode operation. Appendix A lists the firmware commands that affect the Horizontal Rotator. The *Physical Property Measurement System: Firmware Manual* explains in more detail how to control the motor directly from the front panel of the Model 6000 PPMS Controller.

The manually operated rotator consists of the Horizontal Rotator probe and the support plate assembly without a motor.

1.2.2 Electrical Connection to the Model 6000

The motor cable (part number 4084-303) connects the rotator motor to the “P10–Motor” port located on the rear panel of the Model 6000 PPMS Controller. The connections for the “P10–Motor” port are hard wired from the Model 6000 motherboard, providing six signals that consist of four phase lines plus one index signal and one limit signal. The four phase lines provide power to the stepper motor. The index switch at the motor is used as the -10° reference position. The limit signal is permanently deactivated (shorted to ground) and is not used with the motorized Horizontal Rotator option.

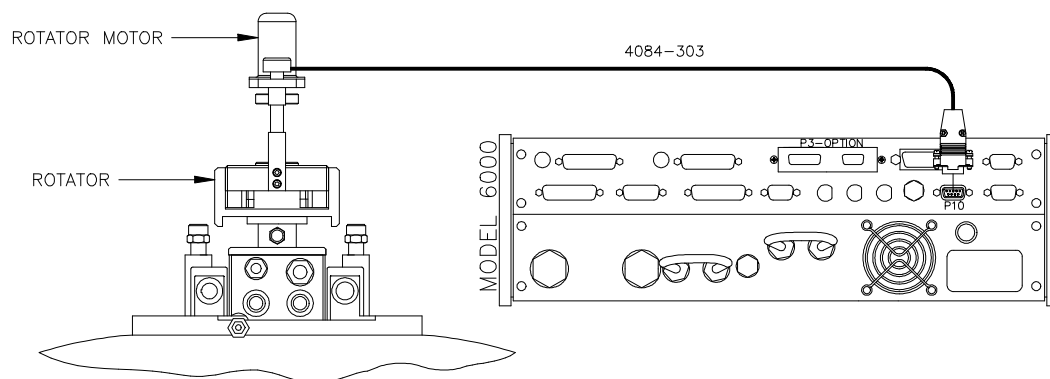


Figure 1-1. Connection Diagram for Horizontal Rotator

1.2.3 Maintenance

The rotator O-rings should be kept clean and lubricated to ensure long life for the PPMS. The O-rings on the top end of the Horizontal Rotator probe (see figure 1-2) should be lubricated with Apiezon M Grease. The O-rings on the bottom of the rotator support plate (see figure 2-6) should be lubricated with silicone vacuum grease. Both greases can be obtained from Quantum Design. Gears, bearings, and other moving parts on the rotator should not come in contact with grease.

Heat sink compound is applied to the top of the rotator thermometer to help keep it in close thermal contact with the sample holder board. Fresh thermal compound should be applied when there is no visible amount of compound left on the thermometer. This thermal compound can also be obtained from Quantum Design.

Table 1-2. Part Numbers for Horizontal Rotator Parts and Accessories

DESCRIPTION	PART NUMBER	ILLUSTRATION
O-ring for Horizontal Rotator Probe	VON-218	Figure 1-2
O-ring for Support Plate	VON2-326	Figure 2-6
Apiezon M Grease (for VON-218)	HM11	
Silicone Vacuum Grease (for VON2-326)	AGV1	
Thermal Compound for Rotator Thermometer	AGT1	
Rotator Support Tool	4084-391	Figure 2-2
Sample Removal Tool	4084-307	Figure 2-3
Resistance Bridge Sample Holder Board	3084-371	Figure 2-1
Universal Sample Holder Board	3084-341	Figure 2-5
AC Transport Sample Holder Board	3084-370	
Horizontal Rotator Kit for ACT Option	4084-308	
• Includes ACT/Horizontal Rotator Probe Cable	3084-517	
• Includes ACT Sample Holder Board	3084-370	

Prices for parts listed in table 1-2 can be obtained by contacting Quantum Design's Customer Service Department. Some parts may also be obtained through other vendors.

1.3 Horizontal Rotator Probe

CAUTION!

The Horizontal Rotator probe is delicate and must be handled with care to avoid denting or bending the stainless steel tube on the probe's exterior. Use caution when working near the intricate components in the rotator transfer case so that you do not damage gears or wiring. Be extremely careful when placing the probe on a flat surface, such as a table, because the probe may easily roll off.

The Horizontal Rotator probe is used to insert the sample into the PPMS sample chamber. A sample holder board is attached to the rotator platform circuit board on the rotator transfer case, and electrical connections to the sample are made through the keyed interface at the bottom of the Horizontal Rotator probe. Refer to figure 1-2.

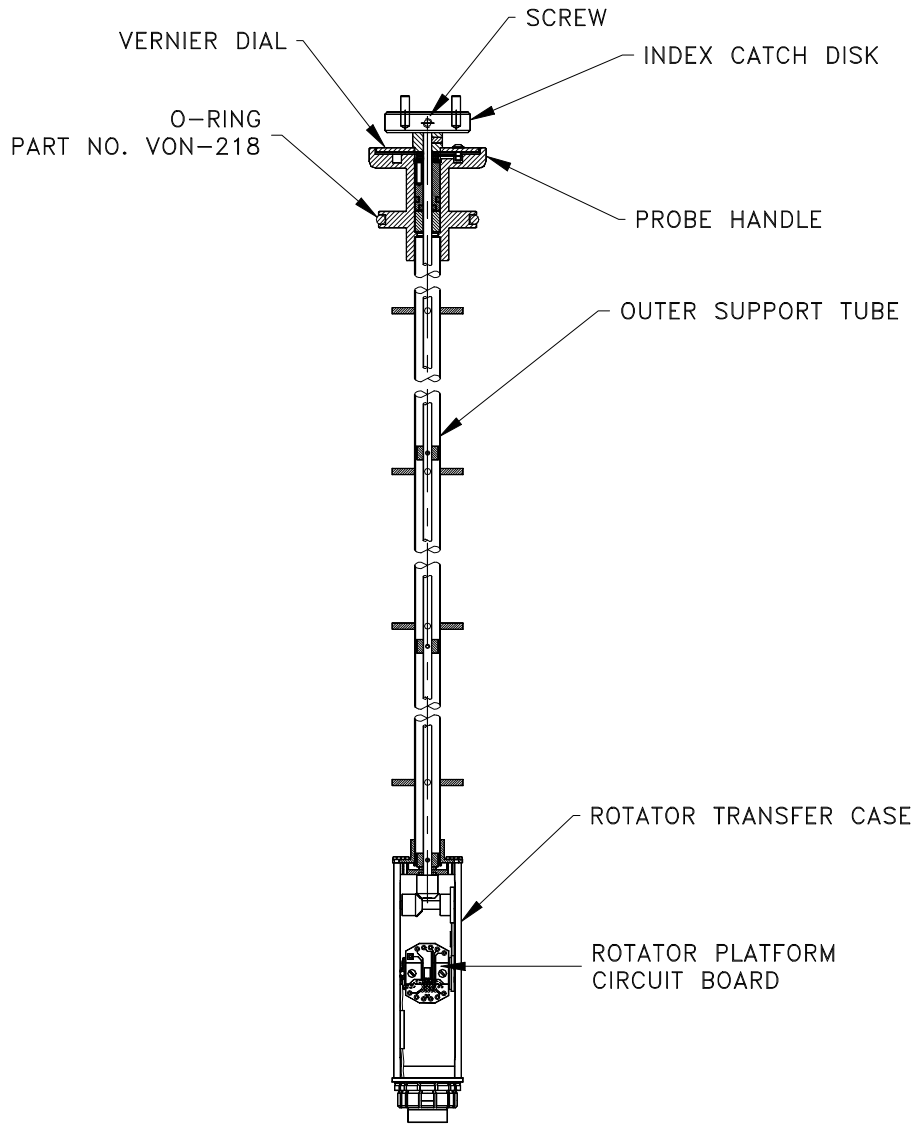


Figure 1-2. Horizontal Rotator Probe

CAUTION!

When it is first removed from the PPMS sample chamber, the Horizontal Rotator probe is usually very cold, and ice typically forms on it, temporarily locking gears and bearings. This is normal. Do not force rotator motion when the rotator is in this condition. The rotator thaws within 1 or 2 minutes, and you may gently work the index catch disk to verify rotation.

CAUTION!

Do not store the Horizontal Rotator probe so that it hangs upside down, and do not rotate the rotator upside down. When the rotator is upside down, the metal pin that prevents the rotator from moving beyond -10° or 370° falls out. This problem can normally be fixed by turning the probe right-side up and rotating it back and forth a few times.

When not in use, the Horizontal Rotator probe should be stored in a vertical position in a hanging rack.

The O-rings on the top end of the Horizontal Rotator probe should periodically be lubricated with grease. Refer to section 1.2.3.

1.3.1 Current Leads

Four copper alloy current leads are wired to the rotator platform circuit board. These leads are connected to contact pads 7, 8, 11, and 12 as labeled on the resistance bridge sample holder board. The remainders of the leads are phosphor-bronze and should only be used for voltage detection.

CAUTION!

The current leads on the rotator (7, 8, 11, and 12) are fine-gauge copper alloy. They are rated for a maximum .500A current. It is possible to output up to 2A current by using the ACT option with the rotator. Depending on temperature and other conditions, this may overheat and melt the rotator wiring. To avoid this situation, limit your output current to .500A while using ACT with the rotator. At room temperature the resistance of the rotator current leads is roughly 0.5Ω .

Note Quantum Design doesn't guarantee the rotator wiring for any current above .500A. However, it is possible to perform experiments with greater current by limiting measurement duration and introducing a cooling period between measurements. Call Quantum Design customer service for details.

1.3.2 Rotator Thermometer

The rotator thermometer is wired to contact pads 3 through 6. User bridge board channel 1 reads the rotator thermometer *unless* the ACT option is in use. When the ACT option is in use, system bridge board channel 4 reads the rotator thermometer.

When the rotator is being used, the settings for user bridge board channel 1 should not be changed from their normal settings for the rotator. These settings are 1000 μ A current limit, 100 μ W power limit, and 10 mV voltage limit. Exceeding these limits can damage the thermometer. Because this thermometer is used by the Model 6000 as the user thermometer that controls system temperature, you

must avoid wiring samples in parallel with the rotator thermometer. Doing so eliminates the ability to accurately read the rotator thermometer and causes erroneous sample measurements.

Heat sink compound is applied to the top of the rotator thermometer. Refer to section 1.2.3.

1.3.3 Changing the Orientation of the Sample Holder Board

To change the angular orientation of the sample holder board that is mounted on the rotator platform circuit board, you use only the index catch disk at the top of the Horizontal Rotator probe. Do not force rotation of the sample holder board itself. The index catch disk is intentionally left loose enough to slip when the rotator has reached its physical limits. This is to prevent damage to the delicate components of the rotator should the motor attempt to move the rotator beyond its physical limits. If the index catch disk is too loose, you may tighten the set screw within this disk by using a 1/16-inch ball driver or Allen wrench. Do not disable this safety disengagement mechanism by gluing or otherwise permanently attaching the index catch disk.

Installation

2.1 Introduction

This chapter contains the following information:

- Section 2.2 summarizes the rotator hardware installation procedures.
- Section 2.3 explains how to mount a sample on a sample holder board.
- Section 2.4 explains how to attach a sample holder board to the rotator probe.
- Section 2.5 explains how to install the rotator hardware into the PPMS.
- Section 0 explains how to download the rotator configuration files.
- Section 0 explains how to ensure proper position calibration.
- Section 0 explains how to remove the rotator hardware from the PPMS.

2.2 Overview of Horizontal Rotator Installation

You must complete the following procedures before you may take measurements with the Horizontal Rotator.

1. Mount the sample on a sample holder board (section 2.3).
2. Attach the sample holder board to the Horizontal Rotator probe (section 2.4).
3. Install the Horizontal Rotator hardware in the PPMS (section 2.5).
4. Download the appropriate rotator configuration file (section 0).
5. Engage the motor and check for proper position calibration (section 0).

If you want to simply attach a different sample to the Horizontal Rotator probe without completely removing the rotator hardware from the PPMS, you may omit steps 4 and 5 above *unless* you perceive problems with rotator operation, in which case these should be your first two lines of action.

Before taking measurements, you should refer to section 3.4, “Measurement Considerations.”

2.3 Mounting the Sample on the Sample Holder Board

The sample is mounted on a sample holder board (see figure 2-1), which is then attached to the rotator platform circuit board on the Horizontal Rotator probe. The sample should be smaller than 8×9 mm so that the sample holder board can be mounted on the rotator platform. The sample holder board makes thermal contact with a thermometer on the rotator platform, allowing close monitoring of the sample temperature when the sample is installed in the PPMS sample chamber. Electrical connections to the mounted sample are made through contact pads on the sample holder board.

To mount a sample on a sample holder board, you (1) secure the sample to the sample holder board and then (2) wire the sample to the sample holder board.

2.3.1 Step 1: Secure the Sample

You may use a variety of techniques, including tapes, glues, greases, and epoxies, to secure samples to the sample holder board. The method you use to secure a sample must be able to withstand the entire temperature range to which the sample will be subjected.

2.3.2 Step 2: Wire the Sample

Electrical contact to the sample is made by using the labeled contact pads on the sample holder board. You typically solder leads to these pads. You may wire samples permanently to the sample holder board and then install the sample holder board on the rotator when you are ready to use the sample.

When the PPMS probe head is connected to the user bridge board, you should wire the samples, which are mounted on the resistance bridge sample holder board, to user bridge board channels 2 and 3 (figure 2-1). The contact pad functions when the probe head is connected to the user bridge board—that is, the contact pad functions for the resistance bridge sample holder board—are identified below in table 2-1.

When the ACT option is activated and the probe head is connected to the Model 7100 AC Transport Controller, the contact pads function differently than they do when the probe head is connected to the user bridge board. Refer to the *Physical Property Measurement System: AC Transport Option User's Manual* for the Horizontal Rotator–ACT wiring arrangement and for detailed information about using the rotator with the ACT option.

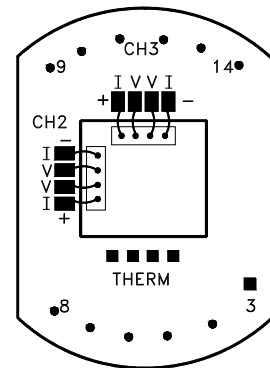


Figure 2-1. Resistance bridge sample holder board with two samples mounted for four-wire resistance measurements.

CAUTION!

Avoid using contact pads 3 through 6 and thus wiring samples in parallel with the rotator thermometer. Wiring samples in parallel with the rotator thermometer invalidates the thermometer readings and causes the PPMS temperature control to malfunction. Refer to section 1.3.2.

Contact pads for the rotator thermometer channel are provided only for the extreme case that you require simultaneous measurements on three channels and want to disconnect the rotator thermometer. Such a course of action is not recommended, and may void the warranty. Contact your Quantum Design service representative before you consider disconnecting the rotator thermometer.

Table 2-1. Standard interconnection table for Horizontal Rotator option. Table shows interconnections between Resistivity option, Horizontal Rotator sample holder board, and thermometer.

RESISTANCE BRIDGE BOARD FUNCTION	P1 PORT ON MODEL 6000	GRAY LEMO CONNECTOR ON PROBE HEAD	RESISTANCE BRIDGE SAMPLE HOLDER	THERMOMETER
Channel 1, I+	5	3	3	I+
Channel 1, I-	18	4	4	I-
Channel 1, V+	6	5	5	V+
Channel 1, V-	19	6	6	V-
Channel 2, I+	7	7	7	
Channel 2, I-	20	8	8	
Channel 2, V+	8	9	9	
Channel 2, V-	21	10	10	
Channel 3, I+	9	11	11	
Channel 3, I-	22	12	12	
Channel 3, V+	10	13	13	
Channel 3, V-	23	14	14	

2.4 Attaching the Sample Holder Board to the Rotator

After the sample is mounted on the sample holder board, you may attach the sample holder board to the rotator platform circuit board that is on the Horizontal Rotator probe. The rotator platform circuit board is located on the rotator transfer case, which is near the bottom end of the Horizontal Rotator probe. Refer to figures 1-2 and 2-5.

The rotator support tool (figure 2-2) and the sample removal tool (figure 2-3) help you avoid damaging rotator hardware when you install and remove the sample holder board. Always use these two hardware components during sample holder board installation and removal.

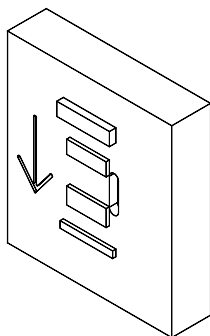


Figure 2-2. Rotator Support Tool

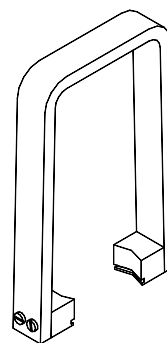


Figure 2-3. Sample Removal Tool

Complete the following steps to attach the sample holder board to the rotator:

1. Set the vernier dial on the Horizontal Rotator probe to 90°.
2. Lay the Horizontal Rotator probe on a flat surface, such as a table, with the chuck key facing upward and the rotator support tool beneath the rotator transfer case. The arrow on the support tool must point toward the handle of the rotator probe (figure 2-4.) This orientation allows the rotator support tool to support the transfer case and prevent the rotator from rolling.

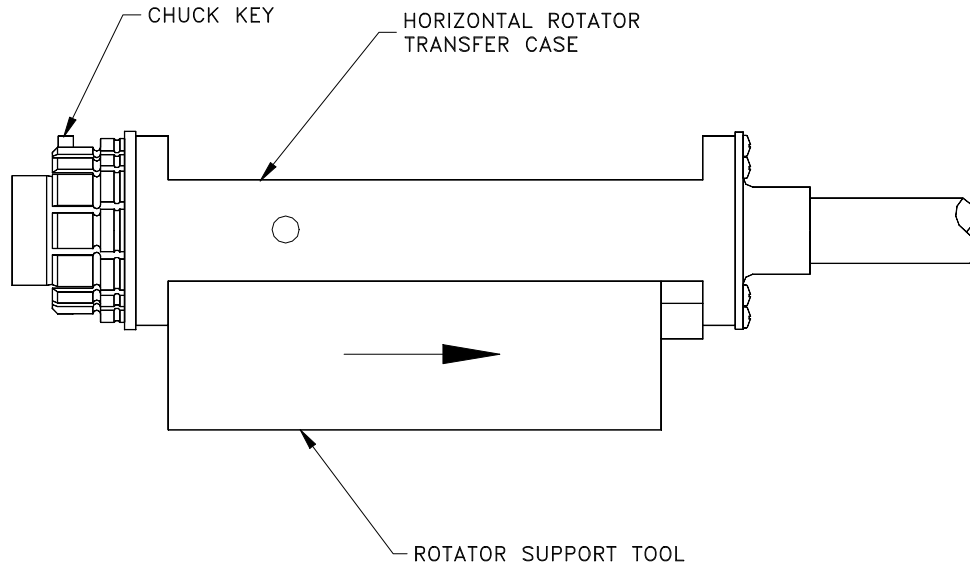


Figure 2-4. Correct Orientation of Rotator Transfer Case on Rotator Support Tool

3. Grasp the sample holder board with the sample removal tool (figure 2-3), and carefully align the pins on the sample holder board with the sockets on the rotator platform circuit board. Refer to figure 2-5 below. Pin #3 is slightly offset from the rest of the pins, so only one orientation of the board is possible. If you force the sample holder board into place with the wrong orientation, you may damage the thin pins on the board.

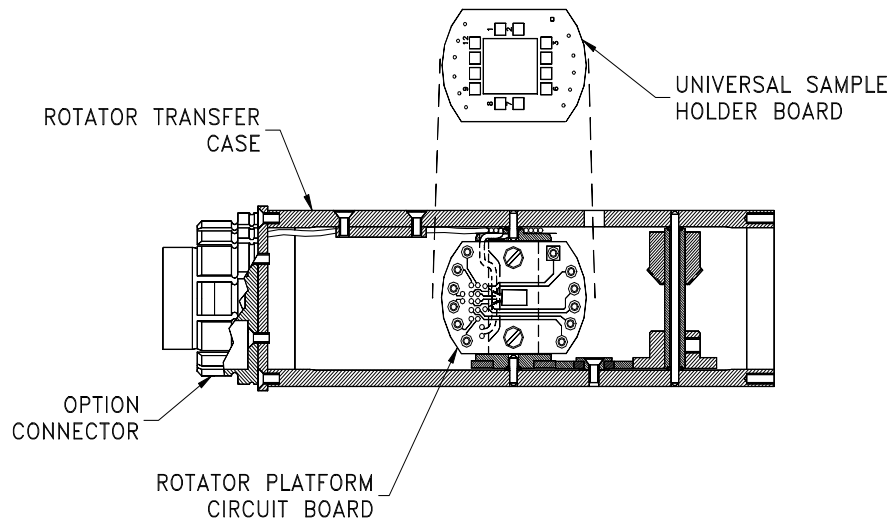


Figure 2-5. Placing the Sample Holder Board on the Rotator Platform Circuit Board

CAUTION!

Use the sample removal tool like a set of tweezers to grasp the sample holder board when you insert the sample holder board on or remove it from the rotator platform. The sample removal tool ensures that even pressure is applied to the sample holder board, therefore keeping the thin pins on the bottom of the sample holder board from bending. Using your fingers to press the sample holder board onto the rotator may bend the pins and damage the electrical connections to the sample.

4. Continue to grasp the sample holder board with the sample removal tool, and then use the sample removal tool to push down the sample holder board completely so that it is immediately on top of the rotator platform circuit board. If the sample holder board is not pushed in with even pressure applied across the board, it may go in crooked and damage the pins. The sample holder board should contact the rotator thermometer with a little thermal compound on top of the thermometer. Once the sample holder board is properly attached, the rotator probe is ready to be installed in the PPMS.

To remove the sample from the rotator probe, follow essentially the same procedure above, being sure you *gently* pull the sample holder board out of place by using the sample removal tool.

Refer to section 1.3.3 to change the angular orientation of a sample holder board that is attached to the rotator probe.

2.5 Installing the Horizontal Rotator Hardware

The following procedure explains how you install the Horizontal Rotator support plate and Horizontal Rotator probe in the PPMS. Section 2.3 explains how you mount a sample on a sample holder board, and section 2.4 explains how you attach the sample holder board to the Horizontal Rotator probe.

CAUTION!

Before you open the sample chamber to the atmosphere, always verify that it is at or above room temperature. If you open the sample chamber while it is below room temperature, condensation and cryopumping of air constituents may occur. This can ultimately cause sample tube deformation and loss of temperature control.

1. Set the PPMS temperature to 298 K, and wait for the temperature to stabilize.
2. Vent the sample chamber continuously.
3. Open the sample chamber and remove any sample puck or PPMS option currently installed in the chamber. Refer to the *Physical Property Measurement System: Hardware Manual* or to the appropriate option manual.

4. Verify that the user bridge cable (part number 3084-003) or the ACT/Horizontal Rotator probe cable (part number 3084-517) is connected (a) to the gray, color-coded port on the PPMS probe head and (b) to other ports as appropriate.

For specific information about the user bridge cable, refer to the *Physical Property Measurement System: Resistivity Option User's Manual*. For specific information about the ACT/Horizontal Rotator probe cable, refer to the *Physical Property Measurement System: AC Transport Option User's Manual*.

5. Place the rotator support plate (figure 2-6) over the PPMS probe head with the centering clip towards the rear of the probe—that is, towards the cable connections.
6. Keep the centering clip up, and firmly squeeze the two flange clamps (figure 2-6) together until they click into place, locking the support plate onto the flange on top of the probe head. You may temporarily remove one or both relief valves from the PPMS helium fill ports to afford easier access for the flange clamps.
7. Flip the centering clip down to lock the two flange clamps against the probe head.

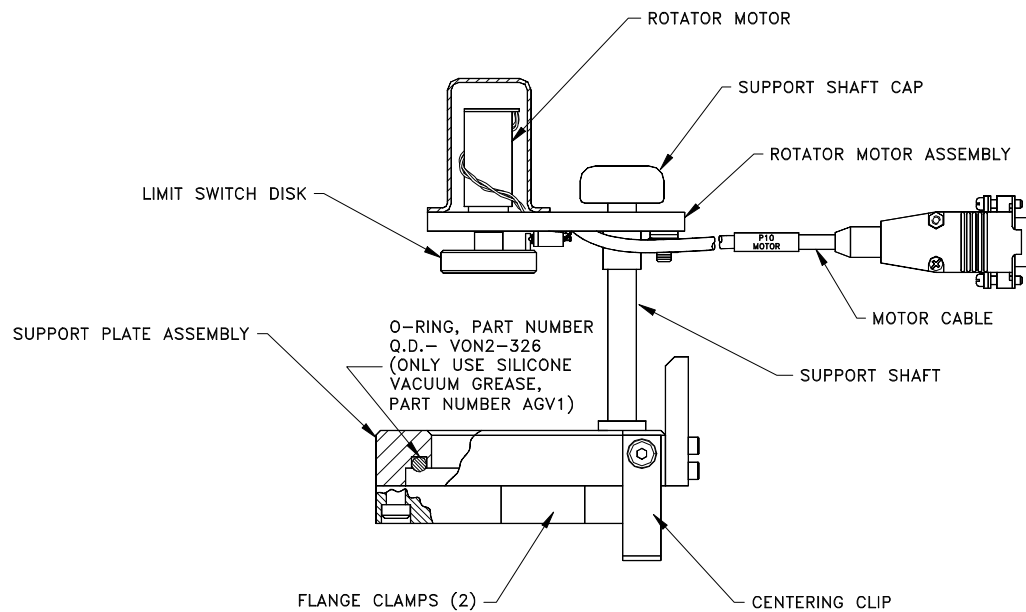


Figure 2-6. Horizontal Rotator Support Plate and Motor

8. Insert the DB-9 connector on the motor cable (part number 4084-303) into the “P10–Motor” port on the rear panel of the Model 6000 PPMS Controller. Tighten the connector screws to hold the DB-9 connector in place.
9. Lift up the rotator motor and swing it to one side of the sample chamber to allow entry into the chamber.
10. Slowly and carefully lower the Horizontal Rotator probe into the sample chamber through the support plate until it just rests at the bottom of the chamber. Guide the rotator probe straight into the sample chamber and do not force it or allow it to bend.

CAUTION!

The Horizontal Rotator probe is delicate and must be handled with care to avoid denting or bending the stainless steel tube on the probe's exterior. Be extremely careful when placing the probe on a flat surface, such as a table, because the probe may easily roll off.

11. Gently rotate the entire Horizontal Rotator probe so that the white mark on the vernier dial faces the front of the PPMS probe. You should feel the keyed connector at the base of the rotator probe drop into place. Firmly push down on the probe to seat it in the connector on the bottom of the sample chamber. It should drop about 0.2 inch (0.5 cm) deeper, sealing the sample chamber, when properly seated. The support plate that you clamped onto the top of the PPMS sample chamber holds the rotator probe in place while sealing the sample chamber.
12. Purge and seal the sample chamber. Installation of the rotator hardware is now complete.

Note Before you use the Horizontal Rotator, you must download the appropriate rotator configuration file as described in section 2.6. You may also want to ensure proper position calibration as explained in section 2.7.

2.6 Downloading the Rotator Configuration Files

After the rotator hardware is first installed or whenever it is reinstalled following use of any other PPMS measurement option, the rotator configuration files supplied with the Horizontal Rotator must be downloaded. Downloading the rotator configuration files ensures that the Model 6000 PPMS Controller uses the proper motor configuration and thermometer calibration.

The Horizontal Rotator configuration file contains the step size information for the rotator motor, the calibration information for the rotator thermometer, and the commands to control the PPMS temperature from the thermometer readings made on the bridge channel. You select the appropriate configuration file for the type of rotator system you are using. The name of each rotator configuration file includes the rotator's serial number. In table 2-2, “###” represents the rotator's serial number.

Table 2-2. Types of Rotator Configuration Files

CONFIGURATION FILE	CABLE FOR BRIDGE BOARD	BRIDGE BOARD READING THERMOMETER CALIBRATION
HR###.CFG	User Bridge Board Cable	User Bridge Board Channel 1
HRACT###.CFG	ACT/Horizontal Rotator Probe Cable	System Bridge Board Channel 4

Complete the following steps to download the rotator configuration files:

1. Insert the disk included with the Horizontal Rotator option into the PC, and select the A: drive.
2. Select the **PPMS 32-bit Tools** icon on the PC desktop, and then run the Romcfg32 utility.
3. Select **Send to PPMS | Send Config** in the main menu.
4. Select the A: drive, which is the drive where the rotator configuration files are located.
5. Locate the rotator configuration file for your system.

- If your system has a standard configuration with the rotator connected to the Model 6000 user bridge board, the appropriate file is `HR###.CFG`, where `###` represents the serial number of the rotator. Refer to table 2-2.
 - If your system uses the special four-way ACT/Horizontal Rotator probe cable, the appropriate file is `HRACT###.CFG`, where `###` represents the serial number of the rotator. Refer to table 2-2.
6. Select the appropriate file, and then click **OK**. After the configuration files are downloaded, the rotator is configured and ready to be used.

For ease of use, you may create a directory called `c:/h_rotat` on the PPMS computer hard drive and copy the configuration files to this directory. Archive the floppy disks for emergency use.

To verify that the configuration file has been read properly, you may check the Model 6000 **STATUS** screen. If the configuration has downloaded properly and the hardware is correctly installed, the **STATUS** screen should display the **User** temperature above the **System** temperature. The user thermometer should initially be a few degrees different than the system thermometer. Configuring the Horizontal Rotator by transmitting the configuration files to the Model 6000 ROM also turns on UserTemp so that the rotator thermometer is used for temperature control.

Note When you remove the Horizontal Rotator probe from the sample chamber, you should turn off the UserTemp thermometer so that the Model 6000 no longer interprets resistance readings on user bridge board channel 1 as temperature readings to be used in system temperature control. Turning off UserTemp is a simple process that is explained below in step 9 of section 2.8.

2.7 Ensuring Proper Position Calibration

To ensure that the position reported by the software matches the actual sample orientation, perform the following steps after you install the Horizontal Rotator probe in the PPMS and download the configuration file.

1. Disengage the motor from the rotator.
2. Use the **Set Position** sequence command to move the motor to the index position and define that position as -10° . Refer to section 3.2.3.
3. Use the **Set Position** sequence command to move the motor to the 0° position. Refer to section 3.2.3.
4. Manually rotate the rotator to the 0° orientation and engage the motor. The rotator probe may require some additional adjustment to achieve proper mating of the limit switch disk on the motor and the index catch disk on the rotator probe. It is acceptable at this point if the vernier dial does not read 0° . However, verify that the index catch disk and the limit switch disk mate concentrically. If these two disks are not concentric, they are not mated properly, but are 180° out of phase.
5. Finalize the position calibration and check for rotation again by using the software to set the rotator to -10° . The motor may move a bit past the limit of rotator motion if the calibration is not already correct. This is intended. After the motor stops moving, set the position to 370° and again wait for the motor to stop moving. After it stops, position calibration is complete. The rotator is ready to begin measurements. For measurement considerations, refer to section 3.4.

2.8 Removing the Horizontal Rotator Hardware

CAUTION!

Before you open the sample chamber to the atmosphere, always verify that it is at or above room temperature. If you open the sample chamber while it is below room temperature, condensation and cryopumping of air constituents may occur. This can ultimately cause sample tube deformation and loss of temperature control.

1. Set the PPMS temperature to 298 K, and wait for the temperature to stabilize.
2. Vent the sample chamber continuously.
3. Disengage the rotator motor from the Horizontal Rotator probe, and pull the rotator probe straight up, guiding it gently out of the sample chamber.

CAUTION!

The Horizontal Rotator probe is delicate and must be handled with care to avoid denting or bending the stainless steel tube on the probe's exterior. Be extremely careful when placing the probe on a flat surface, such as a table, because the probe may easily roll off.

CAUTION!

When it is first removed from the PPMS sample chamber, the Horizontal Rotator probe is usually very cold, and ice typically forms on it, temporarily locking gears and bearings. This is normal. Do not force rotator motion when the rotator is in this condition. The rotator thaws within 1 or 2 minutes, and you may gently work the index catch disk to verify rotation.

Note If you want to attach a different sample to the rotator, do so now. Refer to section 2.4. Then complete steps 8 through 11 in section 2.5 to reinstall the Horizontal Rotator probe in the sample chamber. Do not leave the PPMS sample chamber open to atmosphere for extended periods of time. If you want to completely remove the rotator hardware from the PPMS, complete steps 4 through 9 below.

4. Place the rotator probe in a safe place, preferably hanging vertically.
5. Flip the centering clip on the support plate upwards and release the two flange clamps on the support plate by swinging them out, thus disengaging the support plate from the flange on the PPMS probe head. Refer to figure 2-6.
6. Set the motor aside. You may disconnect it from the "P10-Motor" port on the Model 6000 PPMS Controller, if you like.

7. Install another option or sample puck and blank flange with O-ring.
8. Purge and seal the system.
9. Do the following to set the temperature control back to the system thermometer so that the PPMS does not interpret readings on bridge channel 1 as user thermometer readings:
 - (a) Open **Monitor QD-6000** in the **PPMS 32-bit Tools** folder, or select **Utilities>>Send GPIB Commands** in the PPMS MultiVu interface.
 - (b) Type `USERTEMP 0`.
 - (c) Press <Enter> to execute this command.

You are now ready to use the PPMS for other experiments.

Rotator Motor Operation

3.1 Introduction

This chapter contains the following information:

- Section 3.2 explains how to control the operation of the rotator motor.
- Section 3.3 explains how to set the position configuration parameters.
- Section 3.4 discusses factors to consider when taking measurements.

3.2 Controlling the Rotator Motor

Software commands enable both automated and immediate control of the rotator motor. Commands accessed through the **Instrument>>Motion** option in PPMS MultiVu enable immediate control of any motor connected to the “P10–Motor” port on the Model 6000 PPMS Controller. The **Set Position** and **Scan Position** sequence commands enable automatic motor control from within a PPMS sequence.

The positions to which the sample can move depend on the position configuration parameters and the type of positioner moving the sample. The position configuration parameters for the rotator motor are set by the downloaded rotator configuration file and should never need to be modified unless the speed of the motor must be adjusted or the type of motor being used is changed. Refer to section 3.3.

3.2.1 Status Information

Status information consisting of the index position and the motor’s current and maximum position is displayed at the top of the **Motion Control** dialog box (see figure 3-1 on the following page), which you open by selecting **Instrument>>Motion**. If the motor is configured properly, the index position is identified as -10° and the maximum position is identified as 370° . The lines above and below the position control slide bar also roughly represent the motor’s current position.

The position reported as the motor’s current position and displayed in the **Now at** field in the **Motion Control** dialog box is determined by the number of pulses sent to the motor since its position was last defined; there is no direct feedback from which to obtain the exact sample position. To be certain the reported position matches the true sample orientation, you do the following : (a) set the position to -10° , (b) wait for the software to report it has reached -10° , and then (c) set the position to 370° . After the software reports it has reached 370° , the rotator should be at its upper physical limit (370°).

3.2.2 Movement Control Commands in Immediate Mode

All immediate-mode movement control commands are accessed through the **Instrument>>Motion** menu option in PPMS MultiVu. Selecting **Instrument>>Motion** opens the **Motion Control** dialog box (figure 3-1).

Table 3-1. Immediate-Mode Movement Control Commands

COMMAND	DEFINITION	ACTIVATING
Move To	Motor moves to specified position.	<ul style="list-style-type: none"> • Enter value in Move To text box, then select corresponding Set button. • Drag slide bar button, then select Move To Set button. • Click on slide bar arrows, then select Move To Set button.
Redefine Current Position	System redefines motor's current position as specified position.	Enter value in Redefine Current Position text box, then select corresponding Set button. Motor does <i>not</i> move.
Go to Index	Motor moves to index position.	Select Go to Index button.

As soon as you select a **Set** button or the **Go to Index** button in the **Motion Control** dialog box, the motor begins to move. The system monitors the motor's movement. Status information in the **Motion Control** dialog box identifies each new motor position.

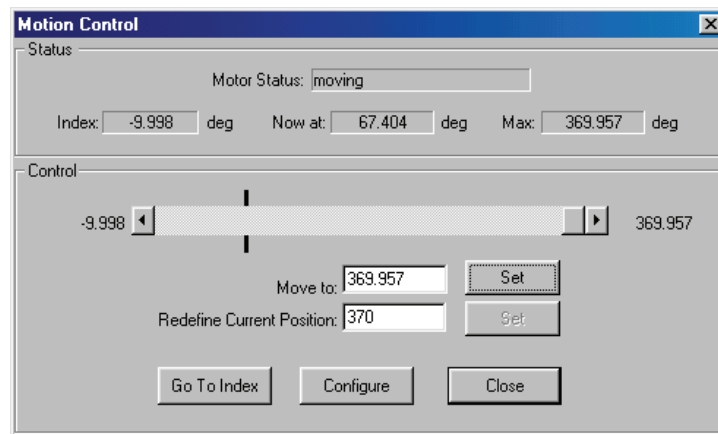


Figure 3-1. Motion Control Dialog Box

3.2.3 Movement Control Commands in Sequence Mode

The **Set Position** and **Scan Position** sequence commands move the motor within a PPMS sequence. **Set Position** moves the motor to single specified position. **Scan Position**, which is a scan command, moves the motor to a series of positions that are within a specified position range. The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses the operation of scan commands in detail.

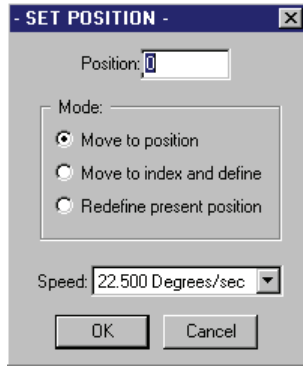


Figure 3-2. **Set Position** Dialog Box
(See Table 3-2)

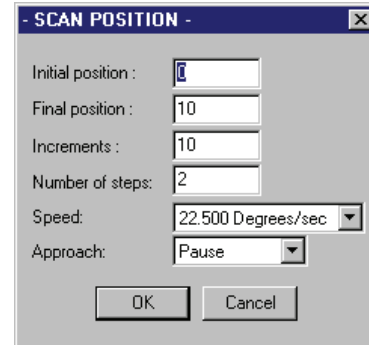


Figure 3-3. **Scan Position** Dialog Box
(See Table 3-3)

Table 3-2. **Set Position** Sequence Command Movement Modes

MOVEMENT MODE	DEFINITION
Move to Position	Motor moves to specified position. Default movement mode.
Move to Index and Define	Motor moves to index switch and system redefines index position with number specified in Position text box in Set Position command. This mode should be used <i>only</i> to redefine index position. With Horizontal Rotator, index position is usually set to -10° .
Redefine Present Position	System redefines motor's present position according to user's specifications.

Table 3-3. Parameters for **Scan Position** Sequence Command

PARAMETER	DEFINITION
Initial Position	First set point in position range.
Final Position	Last set point in position range.
Increments	Number of units between set points.
Number of Steps	Number of set points.
Speed	Rate at which position changes.
Approach	Mode of approaching set points. Pause mode stops motor at each set point. Sweep mode ramps motor at requested speed to final set point and stops at only final set point.

When the Horizontal Rotator is being used to change the sample's orientation, **Measurement** sequence commands and the **Bridge Setup** sequence command are often also included in the sequence along with the **Set Position** or **Scan Position** command. For information about the PPMS measurement option sequence commands, refer to the appropriate option manual. For information about the **Bridge Setup** command, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual*.

The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses sequence operation and all standard system sequence commands in detail. The *Physical Property Measurement System: Commands Manual* also contains information about PPMS sequences.

3.3 Setting the Position Configuration Parameters

Note The position configuration parameters are automatically set by the downloaded rotator configuration file and should never need to be modified unless the speed of the motor must be adjusted or the type of motor being used in the system is changed. Generally, the position configuration parameters are set once during system setup.

The positions to which the sample can move depend on the position configuration parameters and the type of positioner moving the sample. The position configuration parameters are defined in the **Position Configuration** dialog box, which you open by selecting **Instrument>>Motion>>Configure**.

Complete the following steps to verify or change the motor configuration:

1. Select **Instrument>>Motion>>Configure**. The **Position Configuration** dialog box opens. The dialog box lists the values of the position configuration parameters that control how the stepper motor moves the sample.

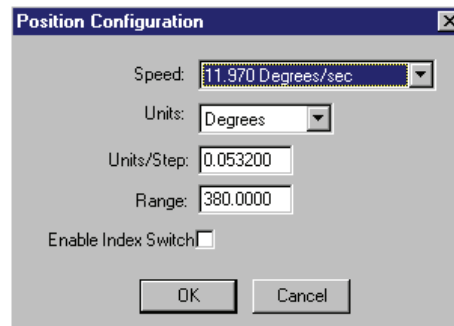


Figure 3-4. **Position Configuration** dialog box displaying correct settings for standard resolution Horizontal Rotator option. When using high-resolution motor, proper number of user units (degrees) in motor steps is 0.00449.

2. Select the speed at which the stepper motor moves the sample.
3. Select the units the system uses to report the sample position. The appropriate selection depends on the configuration of the sample positioner. The allowed values are steps, degrees, radians, millimeters, centimeters, mils, inches, or user defined.
4. Specify the unit distance the sample moves for each step of motion. Units-per-step may be any numeric value.
5. Specify the range of the sample's movement. The range is the maximum distance over which the motor is allowed to move.
6. Enable the index switch, if necessary. If you enable the index switch, the positioner cannot move the sample below the index position. If the index switch is not enabled, it remains a limit, but the sample can move below the index position. By default, the index switch is not enabled. If your system uses a Quantum Design rotator motor, the index switch should *not* be enabled.
7. Select **OK** to enable your selections.

3.4 Measurement Considerations

Most measurement considerations to take into account when designing experiments and writing sequences with the Horizontal Rotator option center around the issue of backlash and the torsional spring found on the rotator. The thermal characteristics and electrical limits of the hardware should also be kept in mind.

The Horizontal Rotator comprises a significant thermal load for the PPMS. The rotator thermometer closely monitors the temperature of the sample(s) on the rotator. However, introducing such a load into the PPMS sample chamber noticeably reduces the speed of the temperature control in the PPMS, especially at higher temperatures (See Section 1.3.1 about Current Leads). Sequences involving temperature changes may take longer than similar sequences run without a rotator installed in the PPMS.

The meshing of the rotator gears is also temperature dependent. Typical behavior is less than 2° of backlash at room temperature (due almost entirely to backlash in the motor), up to 8° of backlash at 150 K, and roughly 6° of backlash at 10 K. While the torsional spring on the rotator is effective at removing backlash at room temperature, its functionality reduces with temperature. Furthermore, if not used conscientiously, the spring can actually cause misleading rotator behavior if the rotator platform circuit board begins to alternately stick and slip during consecutive measurements with small angular changes.

To avoid backlash issues altogether, measurements should always be taken after approaching the sample orientation from the same direction. Since the spring can allow alternate sticking/slipping behavior while it is being unwound, measurements should generally be performed as the spring is being wound tighter. This occurs as the angular orientation is being increased. To achieve the smoothest rotation and most reproducible results, measurements should always be taken on *increasing* angles.

Even with taking such precautions, the correlation of rotator platform orientation to vernier dial reading varies with temperature, up to $\pm 3^\circ$. To fully characterize your rotator at a designated temperature, you may wish to use a Hall sensor, which can be obtained from Quantum Design.

PPMS Firmware Commands

A.1 Introduction

This appendix contains the following information:

- Section A.2 displays the PPMS firmware command tree.
- Section A.3 discusses the firmware commands generally used with the Horizontal Rotator.

A.2 PPMS Firmware Command Tree

The following command tree depicts the Model 6000 PPMS Controller firmware commands that are relevant to the Horizontal Rotator option.

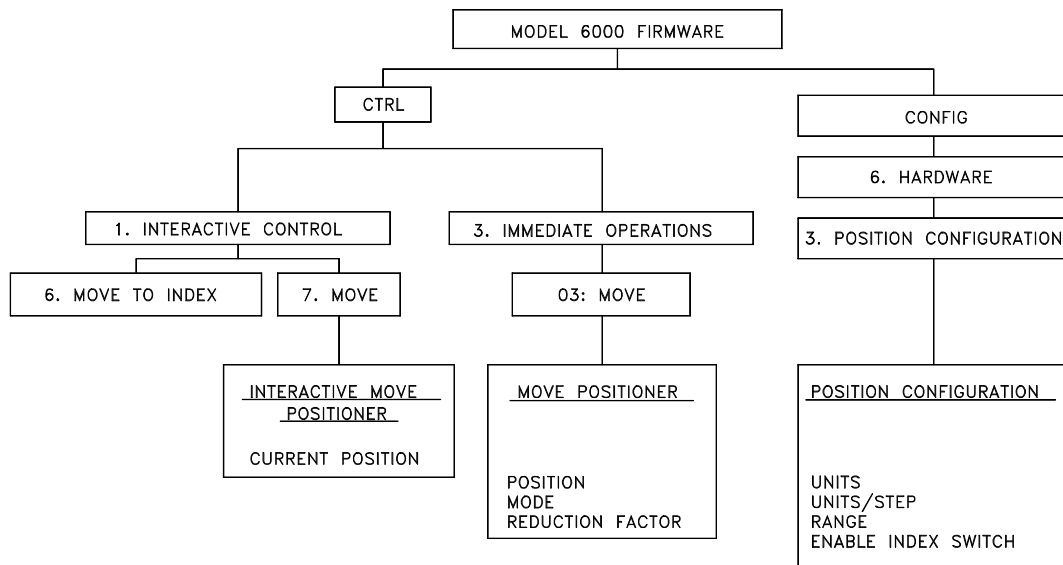


Figure A-1. Model 6000 Firmware Command Tree

A.3 Function of PPMS Firmware Commands

A.3.1 PPMS Firmware Setup Commands

Configuration of the firmware for proper hardware motion control ensures that the PPMS is set up so that measurements may be made properly. Configuration files that are used for the Horizontal Rotator are written into nonvolatile RAM when downloaded to the Model 6000. Alternately, the position configuration can be set from the Model 6000 front panel, as described in the next section.

A.3.1.1 MODEL 6000 POSITION CONFIGURATION COMMANDS

To set the position configuration parameters to their correct values for use with the Horizontal Rotator motor, you access the **Position Configuration** screen in the Model 6000 by selecting **CONFIG>>6. Hardware>>3. Position Configuration**. Set the following parameters within the **Position Configuration** screen in order to configure the motion control variables. Then press **ALT+ENTER** to execute your changes.

Table A-1. **Position Configuration** Parameters for Motion Control Variables

PARAMETER	VALUE
Units	Degrees
Units/Step	0.053200 for standard motor 0.00449 for high-resolution motor
Range	380
Enable Index Switch	No

A.3.2 PPMS Firmware Immediate Operation Command

The Model 6000 **Move** command immediately activates the Horizontal Rotator by controlling the motion of the motor as described in table A-2. To activate the **Move** command, select **CTRL>> 3. Immediate Operations>>03 Move** or press **Enter** while the cursor points to **Position** on the Model 6000 **STATUS** screen. Press **ALT+ENTER** to execute any parameters you select.

Table A-2. Options in **Move Positioner** Screen

SELECT	IN ORDER TO
Position	Specify the position to which the motor moves.
Mode	Set the positioner mode to Normal, Move to Index, or Define Current Position. See table A-3.
Reduction Factor	Select the speed reduction factor.

Table A-3 defines the modes that are available in the **Move Positioner** screen. The screen displays the current angle.

Table A-3. **Move Positioner** Screen Modes

MODE	FUNCTION
Normal	Moves the stepper motor to the specified position.
Move to Index	Moves the motor until the index switch is opened, and then defines the motor position according to the specified value.
Define Current Position	Redefines the motor's current position as the specified position value.

A.3.3 PPMS Firmware Interactive Control Commands

The **Move to Index** and **Move** commands are in the **Interactive Control** menu. You activate **Move to Index** by selecting **CTRL>>1. Interactive Control>>. Move to Index**. You activate **Move** by selecting **CTRL>>1. Interactive Control>>7. Move**.

Move to Index moves the stepper motor in the direction of decreasing angles until the index switch is opened. Then it defines the motor position to be *zero*. For this reason, the interactive **Move to Index** command should not be used to position the rotator motor. The index position when using the rotator is always -10° . The functionality of this command may be changed at a later date so that future versions of the PPMS firmware allow use of this command with the rotator.

The interactive **Move** command moves the motor in single steps as you press the **INCR** or **DECR** keys. Press **ALT+INCR** or **ALT+DECR** to change the position 15 steps at a time. The interactive **Move** screen also displays **limit** and **index** when the limit and index switches are opened.

Interconnection Tables

B.1 Introduction

This appendix contains the following information:

- Section B.2 shows the standard interconnections for the Horizontal Rotator.
- Section B.3 shows the interconnections for the ACT/Horizontal Rotator cable.
- Section B.4 shows the interconnections for the motor.

B.2 Standard Interconnection Table

Table B-1. Standard interconnection table for Horizontal Rotator option. Table shows interconnections between Resistivity option, Horizontal Rotator sample holder board, and thermometer.

RESISTANCE BRIDGE BOARD FUNCTION	P1 PORT ON MODEL 6000	GRAY LEMO CONNECTOR ON PROBE HEAD	RESISTANCE BRIDGE SAMPLE HOLDER	THERMOMETER
Channel 1, I+	5	3	3	I+
Channel 1, I-	18	4	4	I-
Channel 1, V+	6	5	5	V+
Channel 1, V-	19	6	6	V-
Channel 2, I+	7	7	7	
Channel 2, I-	20	8	8	
Channel 2, V+	8	9	9	
Channel 2, V-	21	10	10	
Channel 3, I+	9	11	11	
Channel 3, I-	22	12	12	
Channel 3, V+	10	13	13	
Channel 3, V-	23	14	14	

B.3 Interconnection Tables for the ACT/Horizontal Rotator Probe Cable

The following tables show the standard interconnections between the Model 7100 AC Transport Controller, the ACT sample holder board for the rotator, system bridge board, and thermometer.

Table B-2. Pin Mapping for P2–System Bridge Port on Model 6000

SAMPLE HOLDER BOARD	GRAY LEMO CONNECTOR ON PROBE HEAD	FOUR-PIN LEMO CONNECTOR AT P2 PORT ON MODEL 6000	FUNCTION
3	3	1	Bridge Channel 4 I+ (Rotator Thermometer)
4	4	2	Bridge Channel 4 I– (Rotator Thermometer)
5	5	3	Bridge Channel 4 V+ (Rotator Thermometer)
6	6	4	Bridge Channel 4 V– (Rotator Thermometer)

Table B-3. Pin Mapping for P1–Sample Current Out Port on Model 7100

SAMPLE HOLDER BOARD	GRAY LEMO CONNECTOR ON PROBE HEAD	P1 PORT ON MODEL 7100	FUNCTION
7*	7	1–4	ACT Ch1/Ch2 I+
8*	8	6–9	ACT Ch1/Ch2 I–

* Denoted leads on the Horizontal Rotator are a copper alloy, while the remainder of the leads on the Rotator are phosphor-bronze.

Table B-4. Pin Mapping for P5–Sample Voltage In Port on Model 7100

SAMPLE HOLDER BOARD	GRAY LEMO CONNECTOR ON PROBE HEAD	P5 PORT ON MODEL 7100	FUNCTION
9	9	5 & 7	Channel 2 Va/b+
10	10	6 & 8	Channel 2 V–
11*	11	3	Channel 1 Vb+
12*	12	4	Channel 1 V–
13	13	2	Channel 1 V–
14	14	1	Channel 1 Va+

* Denoted leads on the Horizontal Rotator are a copper alloy, while the remainder of the leads on the Rotator are phosphor-bronze.

B.4 Motor Interconnection Table

Table B-5. Motor interconnection table. Table shows interconnections between motor and P10–Motor port on Model 6000.

DB-9 CONNECTOR	NAME	MOTOR CABLE	DESCRIPTION
9	Index	Black	Index switch
5	Limit Ground	Green*	
4	Limit	Yellow*	Limit wire
2	Phase 1	Red	Coil number 1
7	Phase 2	Violet	
1	Phase 3	Brown	Coil number 2
6	Phase 4	Blue	

* The limit signal wire is shorted to ground on the motor's index switch, as can be seen by examining the underside of the motor assembly.

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



Physical Property Measurement System

Torque Magnetometer Option User's Manual

Part Number 1084-150B

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Third edition of manual completed January 1999.

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U.S. Patents

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)
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.

P.2 Scope of the Manual

This manual describes the PPMS Torque Magnetometer (Tq-MAG) option. This manual describes how you install the Torque Magnetometer, the purpose and function of the Torque Magnetometer, and how you use the hardware and software that are unique to the Torque Magnetometer. **Quantum Design** developed the Torque Magnetometer in collaboration with the **IBM Research Division, Zürich Research Laboratory**, and the **Physik-Institut der Universität Zürich, Switzerland**.




The Torque Magnetometer operates in conjunction with the PPMS Horizontal Rotator or Vertical Rotator option and the user bridge board that is a component of the PPMS Resistivity option. For a detailed discussion of these options, refer to the *Physical Property Measurement System: Horizontal Rotator Option User's Manual*, the *Physical Property Measurement System: Vertical Rotator Option User's Manual*, and the *Physical Property Measurement System: Bridge Board User's Manual* (Quantum Design 1996).

The Torque Magnetometer software operates within the PPMS MultiVu environment and runs on Windows 95/98. This manual assumes you are familiar with the Windows 95/98 operating system.

P.3 Contents of the Manual

- Chapter 1 introduces the Torque Magnetometer option.
- Chapter 2 explains how to calibrate the torque lever.
- Chapter 3 explains how to measure the torque of a sample.
- Appendix A explains how to install the Torque Magnetometer option.
- Appendix B lists standard interconnections for the Torque Magnetometer option.

P.4 Conventions in the Manual

File menu	Bold text distinguishes menus, options, buttons, and panels appearing on the PC monitor or on the Model 6000 PPMS Controller LCD screen.
STATUS	Bold text and all capital letters distinguish the names of keys located on the front panel of the Model 6000 PPMS Controller.
<code>.cal</code>	The Courier font distinguishes characters you enter from the PC keyboard or from the Model 6000 PPMS Controller front panel. The Courier font also distinguishes code and the names of files and directories.
<Enter>	Angle brackets distinguish the names of keys located on the PC keyboard.
<Alt+Enter>	A plus sign connecting the names of two or more keys distinguishes keys you press simultaneously.
	A pointing hand introduces a supplementary note.
	An exclamation point inside an inverted triangle introduces a cautionary note.
	A lightning bolt inside an inverted triangle introduces a warning.

Introduction to the Torque Magnetometer

1.1 Introduction

This chapter contains the following information:

- Section 1.2 explains the function of the Torque Magnetometer option.
- Section 1.3 presents an overview of the hardware used with the Torque Magnetometer option.

1.2 Theory of Operation

The Torque Magnetometer option performs fully automated, angular-dependent measurements of the magnetic torque $\tau = \mathbf{m} \times \mathbf{B}$ experienced by a sample of magnetic moment \mathbf{m} in an applied magnetic field \mathbf{B} . When the torque of the sample is known, the moment of the sample can be determined. The Torque Magnetometer can be used as a complementary tool to DC magnetization and AC susceptibility measurements in order to determine the magnetic properties of materials.

The Torque Magnetometer option features microfabricated, silicon, torque-lever chips. Figures 1-1 and 1-2 illustrate a torque-lever chip. The Torque Magnetometer uses a piezoresistive technique to measure the torsion, or *twisting*, of the torque lever about the lever's symmetry axis. The sample is mounted on a torque-lever chip, and the chip is mounted on a PPMS rotator option. The torque lever twists when a magnetic field is applied to the sample. The system detects the torsion rather than the flexion, or *bending*, of the torque lever in order to minimize gravitational effects that could introduce spurious signals during angular-dependent measurements. A continuous constantan piezoresistive path incorporating two grids patterned on the legs of the torque lever in the region of high stress senses the torque. A Wheatstone bridge circuit that is integrated on the chip measures, to a high degree of sensitivity, the change in resistance, $\Delta(R_1 - R_2)$, of the piezoresistor grids. The change in resistance is due to the mechanical stress produced by the magnetic torque in each leg. The user bridge board in the Model 6000 PPMS Controller measures the voltage across the Wheatstone bridge.

The Torque Magnetometer is fully integrated into the PPMS hardware and software. The Torque Magnetometer uses the full temperature range and field strength that are available to the PPMS, and the Torque Magnetometer software commands are accessed through the PPMS MultiVu application. When the Torque Magnetometer option is active, a command to measure torque may be included in any PPMS sequence.

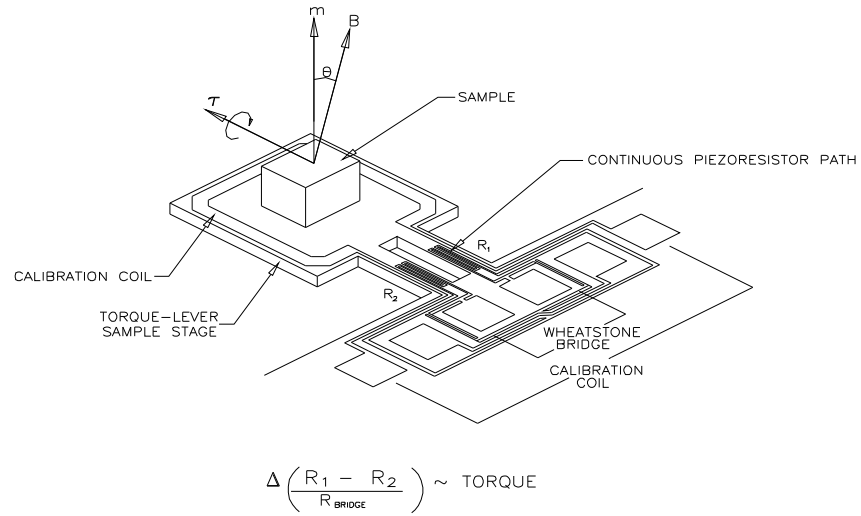


Figure 1-1. Components on Torque-Lever Chip

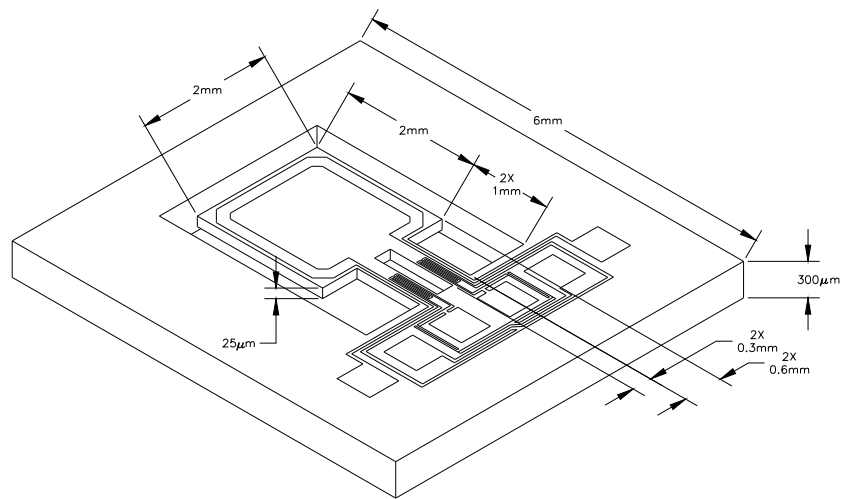


Figure 1-2. Dimensions of Torque-Lever Chip

1.2.1 Measurement Circuitry

The torque-lever chip incorporates a high-sensitivity detection circuit in a Wheatstone bridge configuration. The Wheatstone bridge is directly on the chip; this integrated architecture helps avoid spurious effects that are associated with a split detection circuit.

The Wheatstone bridge is used to measure small changes in the resistance of the piezoresistive path that is on the legs of the torque lever. To measure resistance, the Wheatstone bridge allows the system software to precisely compare the two piezoresistor grids against two other high-precision resistors in the Wheatstone bridge circuit. The two high-precision resistors closely match the resistance of the two piezoresistor grids.

User bridge board channel 2 drives an alternating DC excitation current into the Wheatstone bridge and measures any out-of-balance resistance that can develop across the Wheatstone bridge. By measuring the resistance, the user bridge board can precisely monitor the differential resistance in the piezoresistor grids. The differential resistance is caused by a magnetic torque.

The system software uses a ratiometric measurement of the Wheatstone bridge in order to obtain the highest resolution with a minimum noise level and to reduce temperature hysteresis and magneto-resistive effects. This measurement divides the imbalance of the bridge by the absolute resistance of the bridge. User bridge board channel 3 measures the absolute resistance of the Wheatstone bridge. The system then computes the magnitude of the torque by multiplying the imbalance-to-resistance ratio by the torque coefficient, which is computed during a calibration run. The calibration value is temperature dependent.

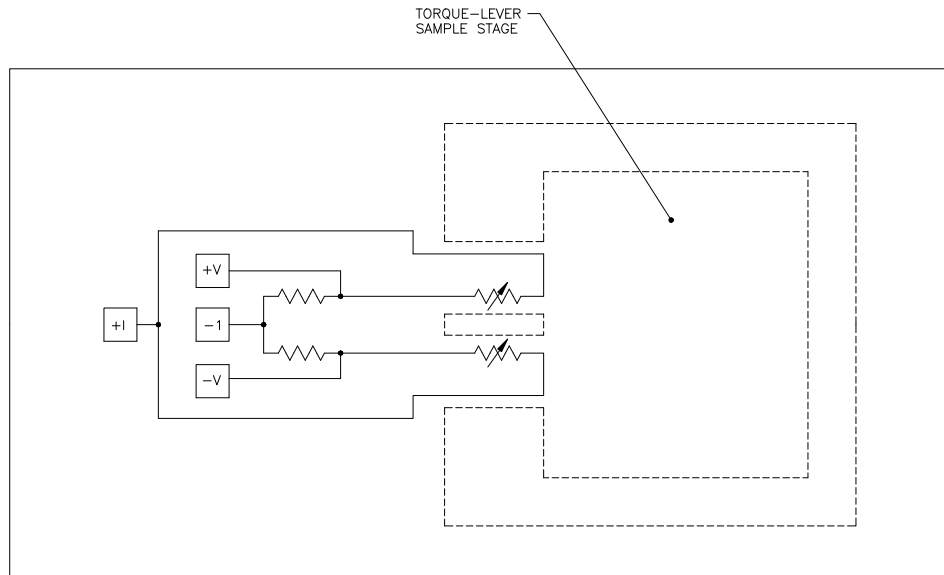


Figure 1-3. Circuit Diagram of Wheatstone Bridge

1.2.2 Calibration Circuitry

The calibration coil, which is a single copper coil loop on top of the torque-lever chip, produces a well-defined magnetic torque τ_{coil} when a small current runs through it. The magnetic torque can be used as a calibration standard for the torsion of the torque lever. Figure 1-1 illustrates the calibration coil.

During a calibration, the torque-lever chip is positioned so that the calibration coil is perpendicular to the external magnetic field. At this orientation, the moment from the coil gives the maximum torque. At a given temperature, a typical current of ± 1.96 mA runs through the coil. The current in the coil produces a well-defined torque that is used as a calibration standard. The system records the change in the resistance ratio. From the change in the resistance ratio, the system calculates a torque coefficient.

The magnetic moment of the calibration coil m_{coil} is defined as

$$m_{coil} = (I_{coil}A_{coil}),$$

and the torque is derived from the equation

$$\tau_{coil} = I_{coil}A_{coil}B \sin\theta,$$

where I_{coil} and A_{coil} are the current through the calibration coil and the area of the calibration coil, respectively, and B is the applied field.

During calibration, the torque coefficient is calculated as

$$C_{\tau} = \frac{2\tau_{coil}}{\Delta \left(\frac{R_1 - R_2}{R_{bridge}} \right)}.$$

In general, special care must be taken to limit the excitation current in the calibration coil in order to avoid heating the torque lever at low temperatures. The software thus limits the current to < 2 mA during a typical calibration.

1.2.3 Sample Type and Size

The Torque Magnetometer measures the magnetization component perpendicular to the applied field (torque $\boldsymbol{\tau} = \boldsymbol{m} \times \boldsymbol{B}$) and is therefore best suited to measure small, anisotropic, ordered materials, such as single crystals and thin films. The Torque Magnetometer can measure spinglasses, paramagnetic materials, and isotropic compounds, but these materials usually yield a negligible torque because \boldsymbol{m} is generally oriented in the same direction as \boldsymbol{B} .

Samples should weigh no more than 10 mg and should have dimensions no greater than $1.5 \text{ mm} \times 1.5 \text{ mm} \times 0.5 \text{ mm}^3$. You should try to limit sample volume in order to avoid torque signals that are greater than 10^{-5} N·m. Signals greater than 10^{-5} N·m produce a nonlinear response in the torque meter. Torque greater than 5×10^{-5} N·m may break the torque-lever chip.

1.2.4 Rotator Options

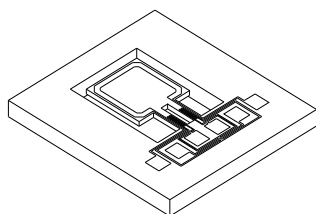
The Torque Magnetometer operates in conjunction with the PPMS Horizontal Rotator or Vertical Rotator option. The rotators enable angular-dependent torque data $\tau(\theta)$ to be gathered in the complete range of environmental conditions that are available in the PPMS probe. From this torque data, useful quantities, such as the axes of easy magnetization and the anisotropy energy, can be determined.

If the PPMS has a transverse magnet configuration, the Torque Magnetometer can operate with either the Vertical Rotator or the Horizontal Rotator option. If the PPMS has a longitudinal magnet configuration, the Torque Magnetometer operates with only the Horizontal Rotator. The *Physical Property Measurement System: Horizontal Rotator Option User's Manual* and the *Physical Property Measurement System: Vertical Rotator Option User's Manual* discuss the rotators (Quantum Design 1996).

1.2.5 User Bridge Board

The Torque Magnetometer operates in conjunction with the PPMS Resistivity option and the user bridge board that is a component of the Resistivity option. User bridge board channel 2 measures the imbalance across the Wheatstone bridge, and user bridge board channel 3 measures the absolute resistance of the Wheatstone bridge. The *Physical Property Measurement System: Bridge Board User's Manual* (Quantum Design 1996) discusses the Resistivity option and the user bridge board.

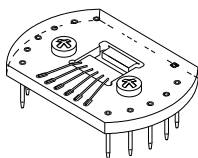
1.3 Overview of System Hardware



Torque-lever chip.

The torque-lever chip (part number 3084-370) includes a lever that provides the mounting platform for the sample. Two legs containing a piezoresistor path that incorporates two grids are used to measure the torque applied to the lever.

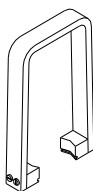
The Torque Magnetometer option includes five chips and a pair of plastic tweezers that is used to pick up the chips.



Platform board assembly.

The platform board assembly (part number 3084-350) connects the torque-lever chip to the rotator platform circuit board.

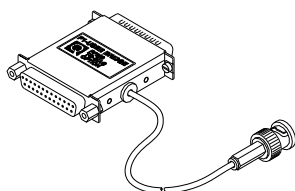
The Torque Magnetometer option includes two platform board assemblies and a small screwdriver that is used to loosen and tighten the screws on the assemblies. Each assembly works with either rotator option.



Sample removal tool.

The sample removal tool (part number 4084-366) holds the platform board assembly while the assembly is installed on or removed from the rotator probe.

The Torque Magnetometer option includes one sample removal tool.



Option interface cable.

The option interface cable (part number 4084-380) connects the user bridge board to the rotator option and limits the current sent to the Wheatstone bridge and the calibration coil. The option interface cable assembly includes the user bridge box. The BNC connector that is attached to the user bridge box provides the connection for the excitation current to the calibration coil.

The Torque Magnetometer option includes one option interface cable.

The Torque Magnetometer option also includes sample boxes of Apiezon N Grease and Apiezon M Grease.

Torque Lever Calibration

2.1 Introduction

This chapter contains the following information:

- Section 2.2 presents an overview of torque lever calibration.
- Section 2.3 explains how to calibrate the torque lever.
- Section 2.4 describes the calibration process.

2.2 Overview of Torque Lever Calibration

The Torque Magnetometer system uses the calibration coil on the torque lever in order to determine the lever's sensitivity at various temperatures. To calibrate the torque lever, the system takes differential measurements, over a specified temperature range, of the change in the resistance of the torque lever at zero torque and at a known positive and negative torque. The system averages the results of each differential measurement to obtain a torque coefficient at each temperature. Then the system saves the torque coefficients to the active calibration table. The system supports sensitivity calibration only in immediate, or manual, mode.

The sensitivity of a torque lever must be calibrated before the torque-lever chip can be used for sample measurements. Calibrating a torque lever should be necessary only once. One calibration table is thus created for each chip.

2.2.1 Calibration Tables

A calibration table contains the torque coefficients and calibration curve for the calibration of the sensitivity of one torque lever. During calibration, the system constantly updates the active calibration table. Every calibration table has a `.cal` file extension.

In a calibration table, the imbalance and resistance of the Wheatstone bridge are expressed in ohms. The imbalance-to-resistance ratio is dimensionless. Torque is expressed in newton-meters. Notice that $1 \text{ N}\cdot\text{m} = 10^{-7} \text{ dyne}\cdot\text{cm}$.

2.3 Calibrating the Sensitivity of the Torque Lever

2.3.1 Apply Grease to the Chip

Use a toothpick or the broken edge of a cotton-tipped applicator to apply a small amount of Apiezon N Grease or Apiezon M Grease to the center of the torque-lever sample stage. Apply only enough grease to hold the sample you will measure. Avoid getting grease on the copper contact springs that touch the torque-lever chip.

The grease you apply is not magnetic, but its weight might influence the twisting of the torque lever and should therefore be taken into account during the calibration of the torque lever's sensitivity.

2.3.2 Mount the Chip on the Platform Board Assembly

1. Position the platform board assembly below a microscope and a strong light.
2. Use the screwdriver included with the platform board assembly to loosen—but not remove—the two brass screws on top of the assembly. Loosen the screws until the backing support plate, which is between the screws, lowers slightly. Figure 2-1 illustrates the components on the platform board assembly.

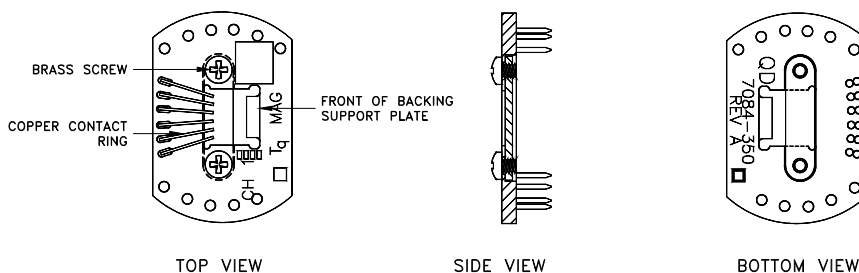


Figure 2-1. Platform Board Assembly

3. Use the plastic tweezers included with the platform board assembly to pick up the torque-lever chip by touching the tweezers to the perimeter of the chip.



CAUTION

The torque-lever chip is delicate and can break easily. Quantum Design recommends that you use only plastic tweezers to handle the chip and that you use the tweezers to pick up only the chip's perimeter. Metal tweezers can more easily bend and break the chip.

4. Use the tweezers to slightly lower the front of the backing support plate, and then position the chip on the backing support plate so that each of the chip's six electrical pads is centered below and touching one of the six copper contact springs on the platform board assembly. Figure 2-2 illustrates the electrical pads on the chip.

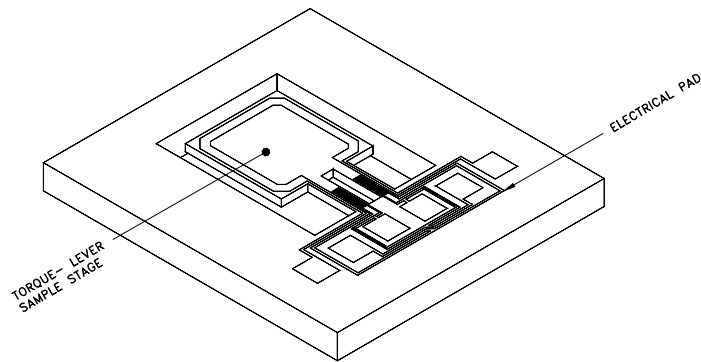


Figure 2-2. Torque-Lever Chip

5. Tilt the platform board assembly sideways to verify that the electrical pads and contact springs are making good electrical contact with each other. If necessary, reposition the chip until all pads and springs are touching.
6. Use the screwdriver to tighten the two brass screws on top of the platform board assembly.

A chip may remain on the platform board assembly permanently. You can use the blank, white space on the top of the platform board assembly to identify which chip is mounted on the assembly.

2.3.3 Mount the Platform Board Assembly on the Rotator

The procedures for mounting the platform board assembly on the Horizontal Rotator probe or the Vertical Rotator probe are similar. The following procedure explains how you mount the platform board assembly on the Horizontal Rotator probe.

1. Place the rotator support tool on a flat surface.
2. Lay the rotator probe on top of the center of the rotator support tool. Position the rotator probe so that the chuck key faces upward and the handle of the rotator probe points toward the arrow on the rotator support tool. Refer to figure 2-3.

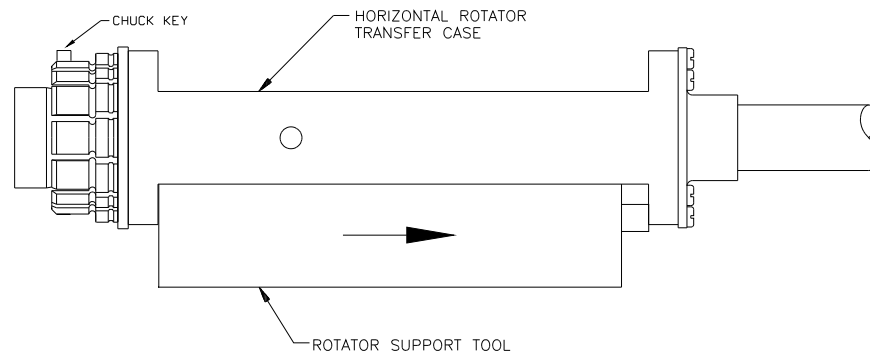


Figure 2-3. Horizontal Rotator Support Tool and Horizontal Rotator Transfer Case

The grooves and ridges on the rotator support tool stabilize the rotator transfer case and prevent the rotator probe from rolling.

- Use the Torque Magnetometer sample removal tool, like you use tweezers, to pick up the platform board assembly. Hold the platform board assembly so that the torque-lever chip faces upward.
The special design of the sample removal tool holds the platform board assembly securely but gently and applies even pressure to the assembly.



Use only the Torque Magnetometer sample removal tool to pick up the platform board assembly when you install the assembly on or remove it from the rotator probe. By applying even pressure, the sample removal tool prevents the pins on the platform board assembly from being bent and damaged.

- Align the pins on the bottom of the platform board assembly with the corresponding sockets on the rotator platform circuit board, and then place the assembly on the circuit board. Use pin #3, which is slightly offset, to help you align the pins and sockets.
- Verify that the platform board assembly touches the rotator thermometer, and verify that all pins and sockets align. If necessary, reposition the assembly. Never force the platform board assembly into place with the wrong orientation.
- Relax the jaws of the sample removal tool, and remove the tool.

2.3.4 Set the Rotator Motor Position

During calibration, the greatest torque is generated when the rotator motor is at 90°. You can use PPMS MultiVu to automatically position the rotator motor. Complete the following steps:

- Select **Instrument** > **Motion** in the PPMS MultiVu interface. The **Motion Control** dialog box opens.

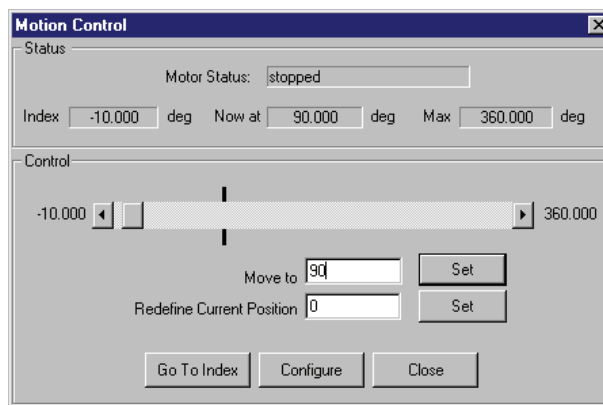


Figure 2-4. **Motion Control** Dialog Box

- Enter 90 in the **Move to** text box.
- Select the **Set** button that is to the right of the **Move to** text box.
- Close the **Motion Control** dialog box.

2.3.5 Insert the Rotator Probe into the Sample Chamber



NOTE

You can use commands in the **Tq-MAG Calibration** dialog box or in the Torque Magnetometer control center to prepare the system for a calibration run. However, the control center does not include the **Calibrate** command. The procedures in sections 2.3.5 through 2.3.10 explain how you use the **Tq-MAG Calibration** dialog box to prepare for and run a calibration.

1. Select **Measure>Tq-MAG Calibration**. The **Tq-MAG Calibration** dialog box opens, and the system starts to check the hardware configuration. Warning messages pop up if the system detects any problems. As soon as the configuration check is complete, the user bridge board channels power up with their default settings.

There are four tabs in the **Tq-MAG Calibration** dialog box. The procedures in the tabs step you through the calibration process. Figure 2-5 illustrates the **Install** tab, which is the first tab in the dialog box.

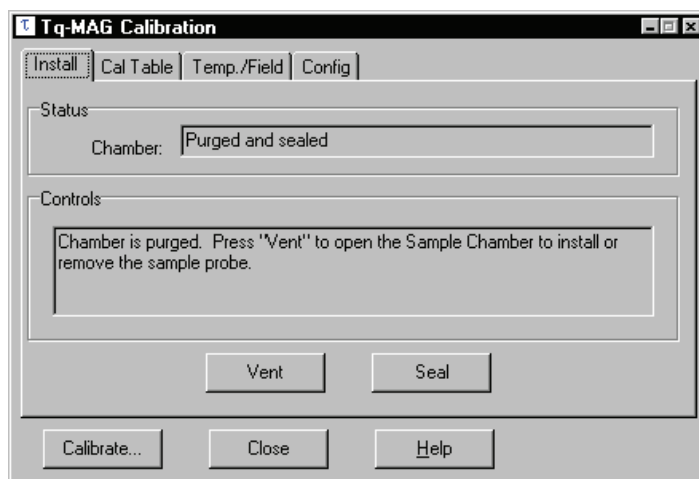


Figure 2-5. **Install** Tab in **Tq-MAG Calibration** Dialog Box

2. Select **Vent**. The sample chamber is flooded with helium unless the system temperature is less than a set minimum temperature.
3. Open the sample chamber when you are prompted to do so.
4. Install the rotator support plate and the rotator motor if these two items are not installed. Refer to sections A.2.4 and A.2.5.
5. Hold the rotator probe so that it is straight, and then slowly and carefully lower it through the opening in the rotator support plate. Do *not* bend the probe while you lower it. Continue lowering the probe until it just rests on the bottom of the sample chamber.
6. Turn the exposed top of the rotator probe until you feel the keyed connector at the base of the probe drop into position.
7. Push down on the rotator probe to seat it in the connector at the bottom of the sample chamber. Seating the rotator probe allows the O-rings in the rotator probe to tightly close the chamber.
8. Select **Seal**. The sample chamber is purged and sealed.

2.3.6 Create the Calibration Table

1. Select the **Cal Table** tab in the **Tq-MAG Calibration** dialog box.

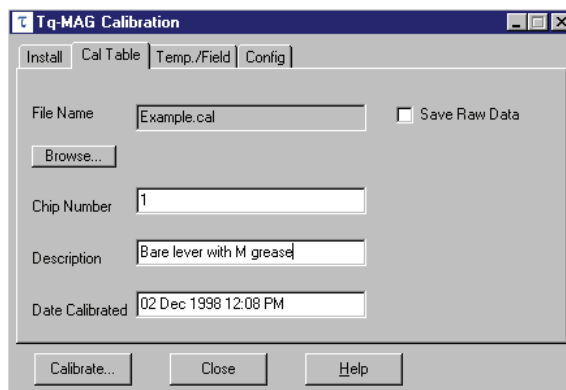


Figure 2-6. **Cal Table** Tab in **Tq-MAG Calibration** Dialog Box

2. Select **Browse**. The **Tq-MAG Create Calibration File** dialog box opens.
3. Use the **File name** text box to enter the name of the new calibration table. In the name, consider including the chip number and a brief description of the sample. The number indicates which chip's calibration data is stored in the calibration table. Both the number and the description help you locate the correct calibration table to use with a sample.
4. Select **Open** and then select **Yes** if PPMS MultiVu asks you whether you want to create the new calibration table.

If you have entered or selected the name of an existing calibration table, PPMS MultiVu tells you that you will be appending data to that table. Cancel the pop-up message, and then enter another name for the calibration table.
5. Use the **Chip Number** text box in the **Cal Table** tab to enter the chip number or another suitable identifier if you want to store this information in the header of the calibration table.
6. Use the **Description** text box to enter a brief description or comment if you want to store this information in the header of the calibration table.
7. Enable the **Save Raw Data** check box if you want to save raw measurement data to a raw data file. Raw measurement data consists of all measurement points in each differential measurement. The file containing raw data can be as large as 1 Mbyte. By default, the system discards raw data in order to save disk space. You should be aware, however, that if a malfunction occurs during calibration, the system can use saved raw data to rebuild the active calibration table.

2.3.7 Set the Temperature and Field

1. Select the **Temp./Field** tab in the **Tq-MAG Calibration** dialog box.

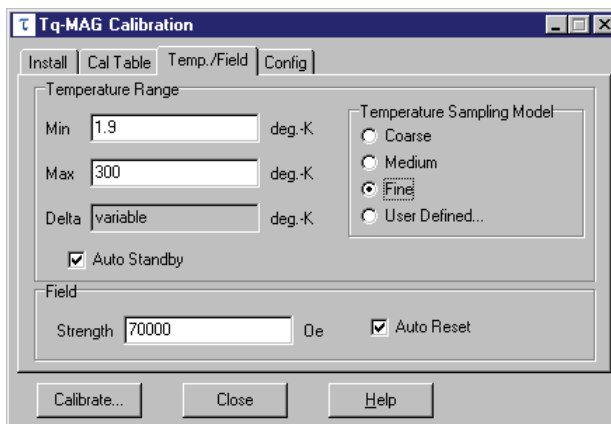


Figure 2-7. **Temp./Field** Tab in **Tq-MAG Calibration** Dialog Box

2. Define the minimum calibration temperature. Use a temperature that equals or is slightly less than the minimum temperature you will use when you measure the torque of the sample. 1.9 K is the default minimum temperature.
3. Define the maximum calibration temperature. Use a temperature that equals or is slightly greater than the maximum temperature you will use when you measure the torque of the sample. 300 K is the default maximum temperature.

The temperature range determines the length of the calibration.

4. Select a temperature sampling model. The temperature sampling model roughly defines the number of temperatures at which the system takes measurements, and it defines delta, which is the increment between temperatures.

The “fine” temperature sampling model is the default sampling model, and it spaces temperatures logarithmically. A user-defined temperature sampling model lets you define the increment between temperatures.

Table 2-1. Temperature Sampling Models and Value of Delta

MODEL	VALUE OF DELTA
Coarse	298.1 K
Medium	50 K
Fine	Variable
User Defined	Variable

5. Disable the **Auto Standby** check box if you do not want the system temperature to enter standby mode when the calibration is complete.
6. Define the field strength for the calibration. Quantum Design recommends using your system’s default maximum field strength because it produces the maximum torque. Depending on the system, the maximum field is 7, 9, or 14 T.
7. Disable the **Auto Reset** check box if you do not want to set the field to zero when the calibration is complete.

2.3.8 Download the Rotator Configuration File

After the Horizontal Rotator or Vertical Rotator option is installed, the rotator configuration file must be downloaded. The rotator configuration file must be downloaded only once. As long as the same rotator is used, downloading the file again is unnecessary.

Complete the following steps to download the rotator configuration file:

1. Select the **Config** tab in the **Tq-MAG Calibration** dialog box.

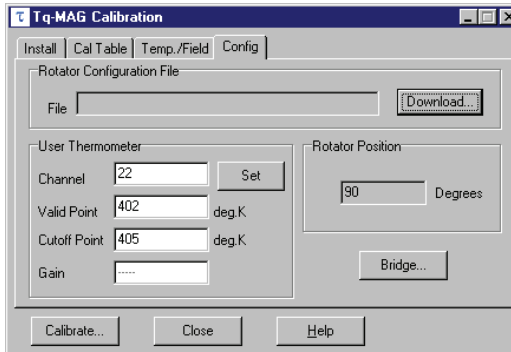


Figure 2-8. **Config** Tab in **Tq-MAG Calibration** Dialog Box

2. Select **Download**. The **Tq-MAG Download Configuration File** dialog box opens.
3. Locate the configuration file that was shipped with your rotator. The file name includes the characters `###.cfg`, where `###` represents the serial number of the rotator.
4. Select the rotator configuration file. The file is downloaded.

The rotator configuration file and the calibration of the user thermometer define the user thermometer's default parameters, which are listed in the **Config** tab. You may change these parameters, but Quantum Design recommends using the default parameters. If you do change any user thermometer parameter, you must select the **Set** button in the **Config** tab in order to enable the new parameter.

2.3.9 Set the User Bridge Parameters

You may change the parameters that affect channels 2 and 3 on the user bridge board. However, Quantum Design recommends using the factory-defined, default user bridge parameters.

Complete the following steps to review or change the user bridge parameters:

1. Select the **Bridge** button in the **Config** tab. The **Advanced** tab opens and becomes the fifth tab in the **Tq-MAG Calibration** dialog box.

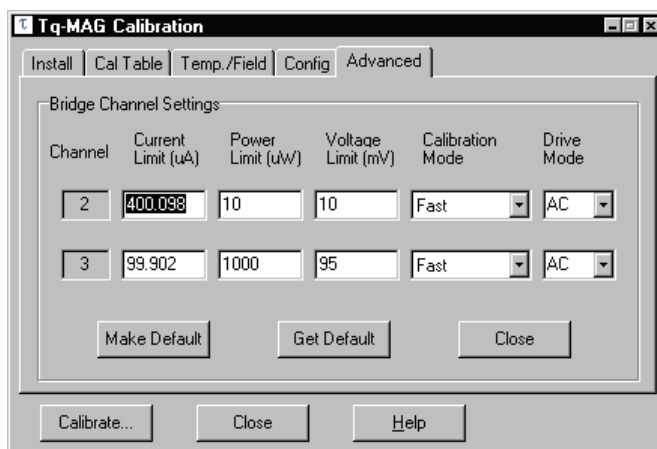


Figure 2-9. **Advanced** Tab in **Tq-MAG Calibration** Dialog Box

2. Modify the parameters of user bridge board channel 2 only if necessary. Table 2-2 lists the default parameters. Channel 2 measures the imbalance of the Wheatstone bridge.
3. Modify the parameters of user bridge board channel 3 only if necessary. Table 2-2 lists the default parameters. Channel 3 measures the total resistance across the Wheatstone bridge.

Table 2-2. Default Parameters for User Bridge Channels 2 and 3

CHANNEL 2		CHANNEL 3	
PARAMETER	DEFAULT VALUE	PARAMETER	DEFAULT VALUE
Current Limit	400 μ A	Current Limit	100 μ A
Power Limit	10 μ W	Power Limit	1000 μ W
Voltage Limit	10 mV	Voltage Limit	95 mV
Calibration Mode	Fast	Calibration Mode	Fast
Drive Mode	AC	Drive Mode	AC

4. Select **Make Default** to make the parameters you have just changed the new default parameters.
5. Select **Get Default** to activate the parameters most recently defined with the **Make Default** command.

2.3.10 Run the Calibration

1. Select the **Calibrate** button, which is at the bottom of the **Tq-MAG Calibration** dialog box.
2. Select **OK** in the pop-up message. If the system is ready for the calibration, the calibration begins immediately. Warning messages pop up if the system detects any problems.

During calibration, the calibration status window pops up and identifies each operation the system performs. Section 2.4 describes the calibration process.

You may open the calibration table if you want to follow the progress of the calibration. Select **File>Open>DataFile**, and then select **All Files** in the **Files of type** drop-down list. Double-click on the name of the calibration table. The graph view of the table opens. Whenever the system computes another torque coefficient, it plots that coefficient, as a measurement point, in the graph.

You may pause or abort a calibration at any time. Simply select **Pause** or **Abort** in the calibration status window. To resume a paused calibration, select **Resume** in the calibration status window.

The system saves calibration status and command information and most calibration error messages to a calibration log file. The log file has the same name as the calibration table, but it has a `.log` extension instead of a `.cal` extension.

3. Wait for the calibration to finish. The specified temperature range determines the length of time the calibration runs. The **Calibration completed** message appears in the calibration status window when the calibration is complete. Another message asks whether you want the system to enter standby mode. If the **Auto Standby** check box is enabled and you do not answer the message within 5 minutes, the system automatically enters standby mode.

When the calibration is complete, you may remove the rotator probe from the sample chamber.

2.4 Description of Calibration Process

The Torque Magnetometer system uses an automated calibration run to calibrate the temperature dependence of the sensitivity of a torque lever. Calibration consists of two phases. First the system measures the resistance and imbalance of the Wheatstone bridge. Then the system takes differential measurements of the torsion of the torque lever.

When the **Calibrate** command is issued, the system downloads a series of calibration sequences to the Model 6000 PPMS Controller. Once the sequences are downloaded, calibration begins. During the first phase of the calibration, the system sets the field to zero and cools the temperature in the sample chamber to the preselected minimum, or base, temperature. While the temperature cools, the system accurately measures the temperature dependence of the resistance and imbalance of the Wheatstone bridge. User bridge board channel 2 measures the imbalance that develops, as a result of thermal stress on the torque lever during the cool-down, in the Wheatstone bridge. User bridge board channel 3 measures the total resistance across the Wheatstone bridge. The system uses the imbalance-to-resistance ratio to minimize temperature and magnetoresistance effects caused by the wires in the rotator and the PPMS probe. The temperature-dependent resistance ratio constitutes a background “baseline” that is subtracted from the resistance ratio during a sample measurement.

When the system reaches the base temperature, the second phase of the calibration begins. The system sets the maximum field strength. The default maximum field is defined by the PPMS magnet. When the field is stable, the sample chamber begins to warm. At preselected temperature increments, the system takes differential measurements of the torque lever’s response to the magnetic torque that is created by the current in the calibration coil. Analog output channel 4 drives a positive and then a negative current of 1.96 mA through the calibration coil. By relating the change in the resistance ratio to the known value of the calibrating torque, the system computes a torque coefficient at a given temperature. The temperature sampling models can vary the spacing between the measurement points.

When the system takes a differential measurement, it saves that measurement’s averaged result, as a torque coefficient, in the active calibration table. The torque coefficient converts resistance measurements into newton-meters. At the end of the calibration, the system computes the best fit of the torque coefficients and applies this best fit to the calibration curve. The calibration curve is used during a torque measurement.

Torque Measurements

3.1 Introduction

This chapter contains the following information:

- Section 3.2 presents an overview of torque measurements.
- Section 3.3 explains how to measure torque in immediate mode.
- Section 3.4 explains how to measure torque in sequence mode.

3.2 Overview of Torque Measurements

To compute the torque of a sample, the Torque Magnetometer system (1) measures the imbalance and resistance of the Wheatstone bridge, (2) subtracts the zero-torque baseline resistance ratio obtained during the first phase of the torque-lever sensitivity calibration, and (3) multiplies the imbalance-to-resistance ratio by the torque coefficient. The system measures the Wheatstone bridge imbalance and resistance by averaging individual readings it has taken at a specified temperature, field, and rotator motor position. The system correctly computes the torque only if the calibration table created for the torque-lever chip is active.

The system supports torque measurements in immediate mode and sequence mode. Immediate mode lets you quickly take a single measurement of only the torque of a sample. Sequence mode lets you take multiple, automated measurements and use both the **Torque** measurement command and standard PPMS sequence commands. Sequence measurement data can also be saved to a data file. Immediate-mode measurement data cannot be saved to a data file.

3.3 Measuring Torque in Immediate Mode

3.3.1 Mount the Sample on the Chip

1. Estimate the maximum moment and torque of the sample. Limit the sample volume in order to restrict the torque magnitude at high fields. The torque meter's response becomes strongly nonlinear when the torque approaches 10^{-5} N·m, and the torque lever may break when the torque exceeds 10^{-5} N·m.
2. Apply a small amount of glue to the center of the torque-lever sample stage only if you are measuring a sample that is strongly magnetic. Apply only enough glue to attach the sample to the stage. Avoid getting glue on the copper contact springs that touch the torque-lever chip.
3. Use tweezers to pick up the sample, then place the sample on top of the sample stage. Position the sample so that it produces the maximum torque and is as close to the center of the stage as possible. Verify that the sample does not touch the calibration coil. If necessary, reposition the sample until it is in the center of the stage and does not touch the coil.

To achieve maximum torque, Quantum Design recommends that you consider the right-hand rule and orient the sample moment so that is perpendicular to the axis of rotation. Figures 3-1 and 3-2 illustrate how you might position a sample on the stage.

If you position a sample as shown in figure 3-1, the sample is positioned well for measurements in which you vary the angle with respect to the applied field. In addition, a sample positioned as shown in figure 3-1 is less likely to be affected by gravity during rotation than a sample positioned as shown in figure 3-2.

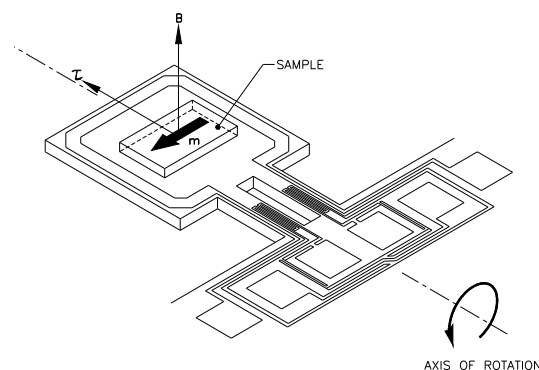


Figure 3-1. Example 1: Sample on Torque-Lever Sample Stage

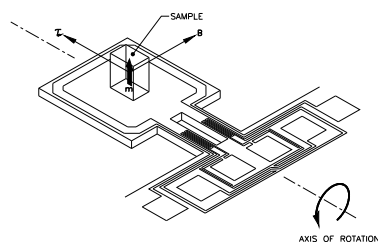


Figure 3-2. Example 2: Sample on Torque-Lever Sample Stage

3.3.2 Mount the Chip on the Platform Board Assembly

1. Position the platform board assembly below a microscope and a strong light.
2. Use the screwdriver included with the platform board assembly to loosen—but not remove—the two brass screws on top of the assembly. Loosen the screws until the backing support plate, which is between the screws, lowers slightly. Figure 2-1 illustrates the components on the platform board assembly.
3. Use the plastic tweezers included with the platform board assembly to pick up the torque-lever chip by touching the tweezers to the perimeter of the chip.



The torque-lever chip is delicate and can break easily. Quantum Design recommends that you use only plastic tweezers to handle the chip and that you use the tweezers to pick up only the chip's perimeter. Metal tweezers can more easily bend and break the chip.

4. Use the tweezers to slightly lower the front of the backing support plate, and then position the chip on the backing support plate so that each of the chip's six electrical pads is centered below and touching one of the six copper contact springs on the platform board assembly. Figure 2-2 illustrates the electrical pads on the chip.
5. Tilt the platform board assembly sideways to verify that the electrical pads and contact springs are making good electrical contact with each other. If necessary, reposition the chip until all pads and springs are touching.
6. Use the screwdriver to tighten the two brass screws on top of the platform board assembly.

A sample may remain on a torque-lever chip permanently, and a chip may remain on the platform board assembly permanently. You can use the blank, white space on the top of the platform board assembly to identify which sample and chip are mounted on the assembly.

3.3.3 Mount the Platform Board Assembly on the Rotator

The procedures for mounting the platform board assembly on the Horizontal Rotator probe or the Vertical Rotator probe are similar. The following procedure explains how you mount the platform board assembly on the Horizontal Rotator probe.

1. Place the rotator support tool on a flat surface.
2. Lay the rotator probe on top of the center of the rotator support tool. Position the rotator probe so that the chuck key faces upward and the handle of the rotator probe points toward the arrow on the rotator support tool. Refer to figure 2-3.

The grooves and ridges on the rotator support tool stabilize the rotator transfer case and prevent the rotator probe from rolling.

3. Use the Torque Magnetometer sample removal tool, like you use tweezers, to pick up the platform board assembly. Hold the platform board assembly so that the torque-lever chip faces upward.

The special design of the sample removal tool holds the platform board assembly securely but gently and applies even pressure to the assembly.



CAUTION

Use only the Torque Magnetometer sample removal tool to pick up the platform board assembly when you install the assembly on or remove it from the rotator probe. By applying even pressure, the sample removal tool prevents the pins on the platform board assembly from being bent and damaged.

4. Align the pins on the bottom of the platform board assembly with the corresponding sockets on the rotator platform circuit board, and then place the assembly on the circuit board. Use pin #3, which is slightly offset, to help you align the pins and sockets.
5. Verify that the platform board assembly touches the rotator thermometer, and verify that all pins and sockets align. If necessary, reposition the assembly. Never force the platform board assembly into place with the wrong orientation.
6. Relax the jaws of the sample removal tool, and remove the tool.

3.3.4 Insert the Rotator Probe into the Sample Chamber



NOTE

Commands in the Torque Magnetometer control center initiate all operations that the system should perform before it runs a measurement. The procedures in sections 3.3.4 through 3.3.8 explain how you use the control center to prepare for an immediate-mode measurement.

1. Select the **Install** tab in the Torque Magnetometer control center. The **Install** tab is the first of six tabs in the control center. The procedures in the tabs step you through normal Torque Magnetometer system operation.
2. Select **Vent**. The sample chamber is flooded with helium unless the system temperature is less than a set minimum temperature.
3. Open the sample chamber when you are prompted to do so.
4. Install the rotator support plate and the rotator motor if these two items are not installed. Refer to sections A.2.4 and A.2.5.
5. Hold the rotator probe so that it is straight, and then slowly and carefully lower it through the opening in the rotator support plate. Do *not* bend the probe while you lower it. Continue lowering the probe until it just rests on the bottom of the sample chamber.
6. Turn the exposed top of the rotator probe until you feel the keyed connector at the base of the probe drop into position.
7. Push down on the rotator probe to seat it in the connector at the bottom of the sample chamber. Seating the rotator probe allows the O-rings in the rotator probe to tightly close the chamber.
8. Select **Seal**. The sample chamber is purged and sealed.



NOTE

You may use the **Data File** tab in the Torque Magnetometer control center to select or create a data file. Immediate-mode torque measurement data is not saved to a data file, but sequence mode data is.

You may use the **Sample** tab in the Torque Magnetometer control center to define the parameters of the sample. All sample parameter information is stored in the selected data file.

3.3.5 Select the Calibration Table

1. Select the **Cal Table** tab in the Torque Magnetometer control center. The tab displays summary data for the active calibration table. The summary data was defined when the calibration table was created.
2. Select **Browse**. The **Tq-MAG Pick Calibration Table** dialog box opens. The dialog box lists the names of the existing calibration tables.
3. Select the calibration table that was created for the torque-lever chip you are using. As soon as you select the calibration table, the table's name appears in the **Table Name** box, which is at the top of the **Cal Table** tab. All data items defined when the table was created appear in the appropriate boxes in the tab.

3.3.6 Calibrate the Baseline Resistance Ratio

Quantum Design recommends that you calibrate the baseline resistance ratio before you measure the torque of the sample. This calibration is optional, but it ensures a more accurate torque measurement than a measurement computed if the system uses the baseline resistance ratio calibration from the torque-lever sensitivity calibration. When a sample is on the torque-lever chip, the baseline calibration changes slightly.

Complete the following steps to calibrate the baseline resistance ratio:

1. Select the **Temperature** tab in the Torque Magnetometer control center.
2. Change the **Expected Temperature Range** of the baseline calibration only if necessary. Quantum Design recommends using the default expected temperature range, which is the range used during the torque-lever sensitivity calibration.

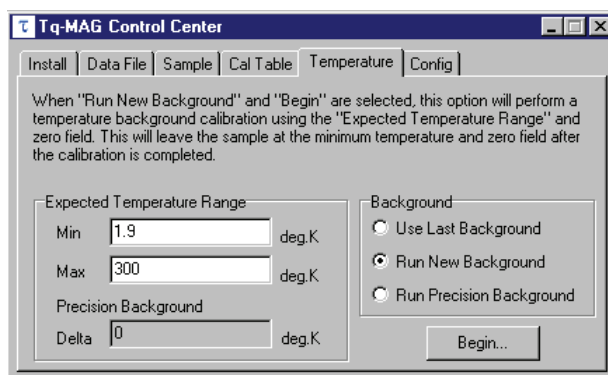


Figure 3-3. **Temperature** Tab in Torque Magnetometer Control Center

3. Verify that the **Run New Background** option is selected.
4. Select **Begin**. The baseline temperature calibration begins. While cooling to the specified minimum temperature, the system measures the imbalance and resistance of the Wheatstone bridge at zero torque and when zero current is in the calibration coil. This latest calibration data is stored in the active calibration table and overwrites the baseline calibration data already in the table. The system then uses the zero-field bridge ratio when it computes the torque of the sample.

3.3.7 Download the Configuration File

After the Horizontal Rotator or Vertical Rotator option is installed, the rotator configuration file must be downloaded. The rotator configuration file must be downloaded only once. As long as the same rotator is used, downloading the file again is unnecessary.

Complete the following steps to download the rotator configuration file:

1. Select the **Config** tab in the Torque Magnetometer control center.
2. Select **Download**. The **Tq-MAG Download Configuration File** dialog box opens.
3. Locate the configuration file that was shipped with your rotator. The file name includes the characters ###.cfg, where ### represents the serial number of the rotator.
4. Select the rotator configuration file. The file is downloaded.

The rotator configuration file and the calibration of the user thermometer define the user thermometer's default parameters, which are listed in the **Config** tab. You may change these parameters, but Quantum Design recommends using the default parameters. If you do change any user thermometer parameter, you must select the **Set** button in the **Config** tab in order to enable the new parameter.

3.3.8 Set the User Bridge Parameters

You may change the parameters that affect channels 2 and 3 on the user bridge board. However, Quantum Design recommends using the factory-defined, default user bridge parameters.

Complete the following steps to review or change the user bridge parameters:

1. Select the **Bridge** button in the **Config** tab. The **Bridge** tab opens and becomes the seventh tab in the Torque Magnetometer control center.
2. Modify the parameters of user bridge board channel 2 only if necessary. Table 2-2 lists the default parameters. Channel 2 measures the imbalance of the Wheatstone bridge.
3. Modify the parameters of user bridge board channel 3 only if necessary. Table 2-2 lists the default parameters. Channel 3 measures the total resistance across the Wheatstone bridge.
4. Select **Make Default** to make the parameters you have just changed the new default parameters.
5. Select **Get Default** to activate the parameters most recently defined with the **Make Default** command.

3.3.9 Measure the Torque

1. Modify the system temperature or field or the position of the rotator motor, if necessary. The system takes the torque measurement at the current temperature, field, and motor position. You may set any position that is available to the rotator, and you may use any field that is available to the PPMS. You should use a temperature that is within the calibration temperature range.

2. Select **Measure** > **Tq-MAG Measure**. The **Tq-MAG Measure** dialog box opens.

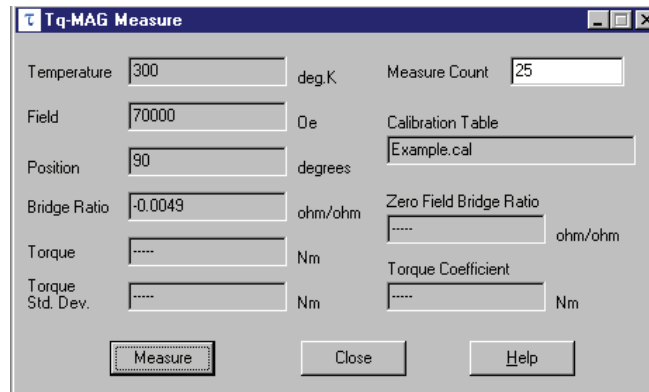


Figure 3-4. **Tq-MAG Measure** Dialog Box

3. Use the **Measure Count** text box to specify the number of readings the system takes and then averages together to compute the imbalance and resistance of the Wheatstone bridge for one sample measurement. Specify a higher number of readings for a sample that is weakly magnetic. Specify a smaller number for a sample that is strongly magnetic or for a relaxation measurement that has short time scales.

The imbalance and resistance of the Wheatstone bridge is the *bridge ratio*.

4. Select **Measure**. The system takes the specified number of readings. During the measurement, you may not use PPMS MultiVu to perform any other function.

As soon as the measurement is complete, the system averages the individual readings to compute the bridge ratio, and then subtracts the zero-field resistance ratio and multiplies the result by the torque coefficient. The system also averages the temperature, field, and motor position used during the measurement. These values appear in the **Tq-MAG Measure** dialog box. The system does not log the measurement to a data file.

Once you insert the torque-lever chip into the sample chamber and select the calibration table, you may take any number of torque measurements in immediate mode.

3.4 Measuring Torque in Sequence Mode

The Torque Magnetometer option includes the **Torque** measurement sequence command. You may include the **Torque** command in a sequence file any number of times in order to automate any number of torque measurements. Within the same sequence, you may use any standard PPMS system sequence commands or advanced sequence commands to control PPMS operation as necessary.

Sequence-mode torque measurement data, unlike immediate-mode torque measurement data, can be saved to a data file. The **Data File** tab in the Torque Magnetometer control center displays the name and file location of the data file that is selected to store torque data. You can use the tab to select or create another data file. The **Browse** button in the **Data File** tab opens the **Tq-MAG Select Datafile** dialog box, which can display the names of all existing data files.

Before the system can measure torque in sequence mode, you must install a sample, define parameters as necessary, and select a calibration table—just as you would if you were preparing to run a measurement in immediate mode. Sections 3.3.1 through 3.3.8 explain the procedures you complete before you run a measurement in immediate mode.

The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* (Quantum Design 1998) discusses sequence mode, sequence files, and sequence execution in more detail.

3.4.1 Torque Measurement Sequence Command

To include the **Torque** command in a sequence, you simply double-click on the **Torque** command name in the sequence command bar. You can open the sequence command bar whenever a sequence file is open. The **Torque** command is in the **Measurement** command group.

When you double-click on **Torque**, the **Torque** dialog box opens. To add the command to a sequence, you simply select the **OK** button that is near the bottom of the **Torque** dialog box. Notice, however, that you may use the dialog box to select parameters that are measured when the torque is measured. You may also specify the number of readings that are taken and then averaged together to compute one sample measurement. Several default parameters are automatically selected in the **Torque** dialog box; refer to figure 3-5.

The **Torque** command also includes a filter-data option, which prompts the system to filter out extremely unusual data readings. By default, the **Filter Data** check box is selected.

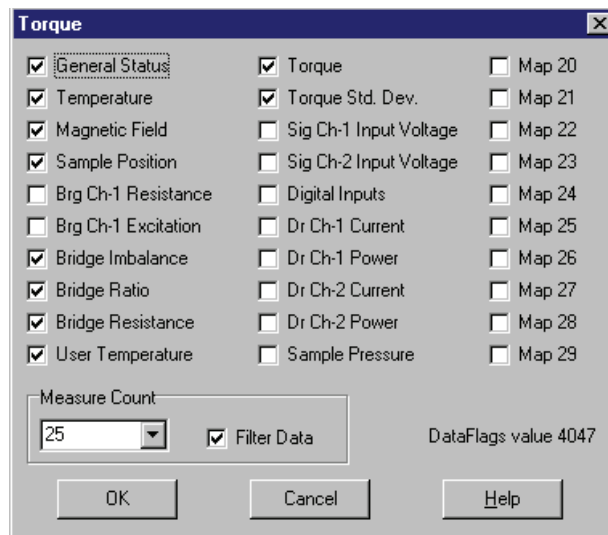


Figure 3-5. Torque Dialog Box

Installation Instructions

A.1 Introduction

This appendix contains the following information:

- Section A.2 contains procedures for installing the Torque Magnetometer option.

A.2 Installing the Torque Magnetometer Option

A.2.1 Install the Option Software

1. Insert Torque Magnetometer Disk 1 into the PC.
2. Select the A: drive.
3. Select `Setup.exe`.
4. Complete all on-screen instructions the InstallShield wizard prompts you to perform.
5. Select **Utilities** > **Activate Option**. The **Option Manager** dialog box opens. The dialog box lists all options that are available on your PPMS and indicates whether each option is active.
6. Deactivate any active measurement option. Click on the option name in the **Active Options** panel, and then select **Deactivate**. The option name moves to the **Available options** panel.

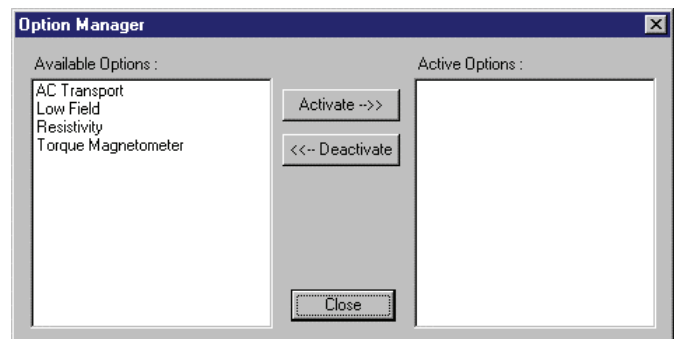


Figure A-1. **Option Manager** Dialog Box

Only one measurement option may be active at a time. An error message pops up if you try to activate a second measurement option. More than one non-measurement option may be active at a time. A measurement option and any number of non-measurement options may also be active simultaneously.

7. Activate the Torque Magnetometer option. Click on **Torque Magnetometer** in the **Available Options** panel, and then select **Activate**. The option name moves to the **Active Options** panel. The Torque Magnetometer control center opens as soon as the Torque Magnetometer software is integrated into PPMS MultiVu. The **Option Manager** dialog box automatically closes.

A.2.2 Verify the User Bridge Board Installation

Verify that the user bridge board is installed in the Model 6000 PPMS Controller and properly connected to the PPMS probe head and the ports on the Model 6000. The *Physical Property Measurement System: Hardware Manual* (Quantum Design 1998) discusses the proper usage of all PPMS ports and connections.

A.2.3 Install the Option Interface Cable

1. Disconnect the user bridge cable from the “P1–User Bridge” port that is on the rear of the Model 6000. The *Physical Property Measurement System: Hardware Manual* (Quantum Design 1998) illustrates all ports on the Model 6000.
2. Attach the female connector of the user bridge cable to the male connector on the option interface cable. Use the provided screws to hold the connectors in place.
3. Attach the female connector of the option interface cable to the “P1–User Bridge” port. Use the provided screws to hold the connectors in place.
4. Attach the BNC connector, which extends from the side of the option interface cable’s user bridge box, to the “A4–Analog Outputs” port that is on the rear of the Model 6000.

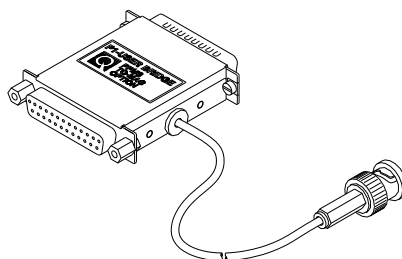


Figure A-2. Option Interface Cable for User Bridge



Remove the option interface cable when the Torque Magnetometer option is not in use. If the option interface cable is used with other PPMS options, the excitation current generated by the BNC connector destroys the accuracy of any measurements that are taken.

A.2.4 Install the Rotator Support Plate

The procedures for installing the Horizontal Rotator hardware or Vertical Rotator hardware are similar.

1. Open the sample chamber. Refer to the *Physical Property Measurement System: Hardware Manual* (Quantum Design 1998).
2. Remove any puck or option components that are in the sample chamber. Refer to the *Physical Property Measurement System: Hardware Manual* or to the appropriate PPMS option manual.
3. Hold the rotator support plate so that its centering clip faces the rear of the probe head, and then place the rotator support plate over the top of the probe head. Verify that the centering clip faces upward.

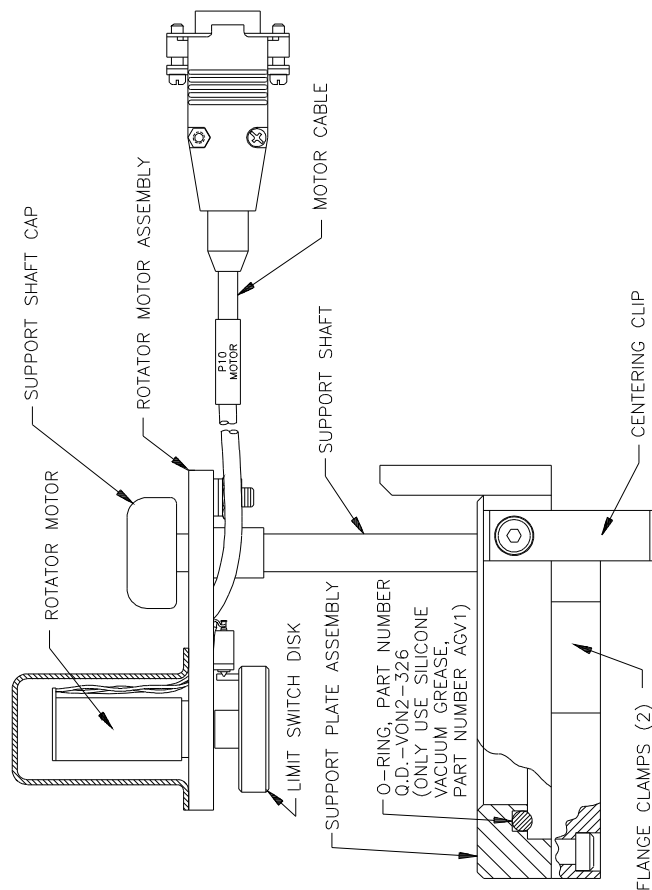


Figure A-3. Rotator Motor and Rotator Support Plate

4. Squeeze the two flange clamps on the rotator support plate together until they click into place and thus lock the support plate onto the top of the probe head. You may temporarily remove one or both of the relief valves from the PPMS helium fill ports in order to access the flange clamps.
5. Flip the centering clip downward to capture the two flange clamps and to lock the rotator support plate against the probe head.

A.2.5 Install the Rotator Motor

1. Attach the DB-9 connector of the rotator motor cable to the “P10–Motor” port on the rear of the Model 6000. Tighten the connector screws.
2. If you are installing the Horizontal Rotator, lift up the rotator motor and swing it to one side of the sample chamber in order to allow entry into the chamber.

Torque Magnetometer Interconnections

B.1 Introduction

This appendix contains the following information:

- Section B.2 lists standard interconnections for the Torque Magnetometer option.

B.2 Standard Interconnections

Figure B-1 illustrates the standard interconnections between the rotator platform and the platform board assembly.

Table B-1 lists standard interconnections between the PPMS probe head, the Model 6000 PPMS Controller, and the Torque Magnetometer when the standard user bridge cable (part number 3084-003) and the Torque Magnetometer option interface cable (part number 3084-380) are in use.

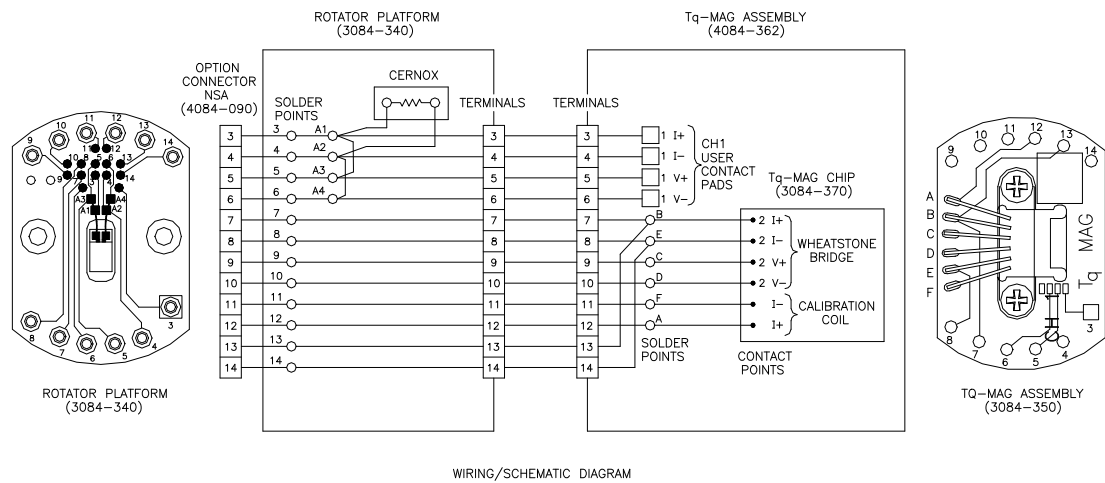


Figure B-1. Wiring Diagram for Platform Board Assembly

Table B-1. Standard Torque Magnetometer Interconnections

OPTION INTERFACE CABLE	GRAY LEMO CONNECTOR ON PROBE HEAD	PLATFORM BOARD ASSEMBLY	TQ-MAG FUNCTION
5	3	3	Channel 1, I+ Rotator thermometer
18	4	4	Channel 1, I- Rotator thermometer
6	5	5	Channel 1, V+ Rotator thermometer
19	6	6	Channel 1, V- Rotator thermometer
7	7	7	Channel 2, I+ Wheatstone bridge resistance imbalance
20	8	8	Channel 2, I- Wheatstone bridge resistance imbalance
8	9	9	Channel 2, V+ Wheatstone bridge resistance imbalance
21	10	10	Channel 2, V- Wheatstone bridge resistance imbalance
9	7	7	Channel 3, I+ Absolute Wheatstone bridge resistance
22	8	8	Channel 3, I- Absolute Wheatstone bridge resistance
10	13	13	Channel 3, V+ Absolute Wheatstone bridge resistance
23	14	14	Channel 3, V- Absolute Wheatstone bridge resistance
BNC connector	11	12	Calibration coil, I+
BNC connector	12	11	Calibration coil, I-

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



VersaLab

Sample-Queue Option User's Manual

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5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.
- Section P.5 describes the safety guidelines and regulatory information in the manual.
- Section P.6 contains disposal information.
- Section P.7 contains information on how to contact your Quantum Design service representative.

P.2 Scope of the Manual

This manual discusses the Quantum Design Sample Queue exchanger for the Physical Property Measurement System (PPMS) VersaLab™. It contains information about basic functionality, describes the system hardware, and the control software.

The Sample Queue is Quantum Design's new, easy to use, completely automated solution for increasing the throughput of magnetic measurements in the VersaLab™ platform. Its compact, integrated design drastically reduces sample loading time by automatically exchanging samples without user intervention. This makes the Sample Queue ideal for optimizing throughput for laboratories running standard Vibrating Sample Magnetometer (VSM) and AC magnetometry (ACMS II).

P.3 Contents of the Manual

- Chapter 1 provides an overview and theory of the operation of the Sample Queue.
- Chapter 2 provides an overview of the Sample Queue option installation.
- Chapter 3 describes details of the Sample Queue hardware.
- Chapter 4 describes Sample Queue software.
- Appendix A describes electrical pin outs and the Sample Queue connections.

P.4 Conventions in the Manual

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

CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.




WARNING!

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




ELECTRIC SHOCK!

This symbol signals electrical hazards that could result in bodily harm, or loss of life. Used at all accessible 200-230 V and 380-408 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).



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.



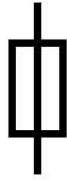
STANDBY SYMBOL

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 users 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.



This symbol signals information on **fusing**.

P.5 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 Sample Queue and reach its best performance.

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse or improper operation of the system.



WARNING!

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.5.1 Inspection for Damage

The Sample Queue measurement option 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.



WARNING!

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.5.2 Electricity



WARNING!

High voltage is supplied to the compressor making a shock hazard possible if inadequate safety procedures are followed.

- In case of emergency, switch the power off at the back of the system console or unplug the main power cords from the wall power outlets.

Observe the following safety guidelines when you use your system:

- 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. To prevent electrical shock, disconnect the compressor from the power before you install it, adjust it, or service it.
- Keep electrical cords in good working conditions, and replace frayed and damaged 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.
- In general, keep liquids away from the PPMS and Sample Queue equipment.
- Keep the PPMS and Sample Queue away from radiators and heat sources. Be sure to follow circuit breaker specifications outlined in the PPMS manual.

P.6 Disposal Information

The Sample Queue is excluded 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 Sample Queue 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.6.1 RoHS Statement

The Sample Queue is explicitly excluded from the Recast RoHS Directive in Article 2(4)(f) which covers: “*equipment specifically designed solely for the purpose of research and development only made available on a business- to-business basis*”.

P.6.2 WEEE Statement

WEEE Statement: The Sample Queue 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.

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

<i>Component Description / Location</i>	<i>Identifying Photograph/Diagram</i>
Sample Queue Module (QD P/N: 3101-070)	

P.7 Contacting Quantum Design

If you have trouble with your Sample Queue or your system, please contact your local Quantum Design service representative for assistance. See www.qd-international.com for the information about your local representative. You will be asked to describe the problem, the circumstances involved, and the recent history of your system.

Introduction

1.1 Introduction

This chapter contains the following information:

- Section 1.2 gives an overview of the design elements and of the operation for the Sample Queue option.

1.2 Overview of the Sample Queue

The Quantum Design Sample Queue option for the VersaLab™ is a versatile turn-key solution for streamlining the testing of Vibrating Sample Magnetometer (VSM) and AC Susceptibility and DC magnetization (ACMS II) samples. The revolving Sample Carousel allows for up to six sample rods to be sequentially loaded and the samples tested in the VSM (ACMS II) system automatically.

There are four main elements that comprise the Sample Queue Hardware system (see Figure 1-1): 1) the sample carousel, 2) the vertical translator and carriage arm, 3) the sample chamber gate valve door and, 4) the control module electronics.

The sample carousel function is to store, organize and sequentially rotate up to six sample rods into position for the vertical translator and carriage arm to pick up. The Sample Queue allows the user to randomly access, load and unload any sample rod into the sample carousel.

The vertical translator and carriage arm function is to pick up a selected sample rod from the Sample Carousel and install it into the VersaLab™ sample chamber.

The sample chamber gate valve door opens the VersaLab™ sample chamber when a selected rod needs to be installed or uninstalled from the sample chamber. Once a selected sample rod is installed in the linear motor the gate valve closes and hermetically seals the chamber during measurements.

The Sample Queue is a flexible automated sample management system that includes all of the electronics needed to communicate with the VersaLab™ host computer and drive the Sample Queue operations. In addition, the Sample Queue software option, enables the user to control the Sample Queue system through immediate and sequence commands available in MultiVu.

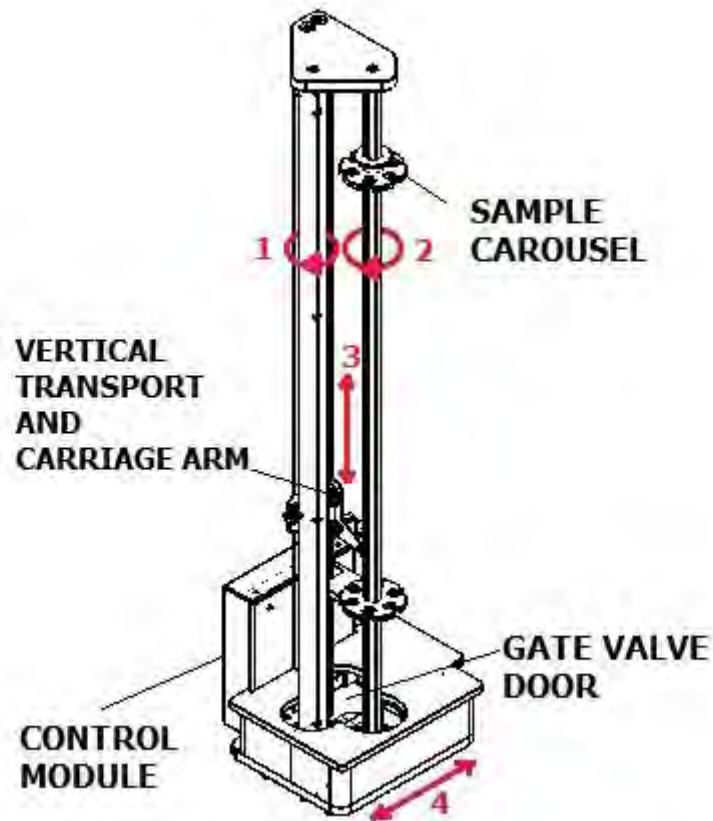


Figure 1-1. Overview of the Sample Queue: 1) Carriage arm drive, 2) Sample Carousel, 3) Vertical Translator arm drive and, 4) Gate Valve drive.

The rest of this chapter describes the major elements of the Sample Queue system. After reading this chapter, be sure to read the rest of the manual in order to learn more about Installing and Removing the Sample Queue option (Chapter 2), details of the Sample Queue Hardware (Chapter 3), and operation of the software interface (Chapter 4). You might also want to consult the appendix to learn about electronics pin out and maintenance procedures.

1.2.1 The Sample Carousel

The Sample Queue carousel is a rotating circular rack that holds up to six sample rods. The sample carousel is comprised of an upper sample rod tray and a lower indexing circular plate. Each of the six slots on the rack is marked with a number on the rack rotating post.

Both VSM and ACMS II sample rods will be captured and mechanically stable when loaded into the sample carousel tray (see Figure 1-2). The sample carousel hub aligns the sample rods within the sample carousel.

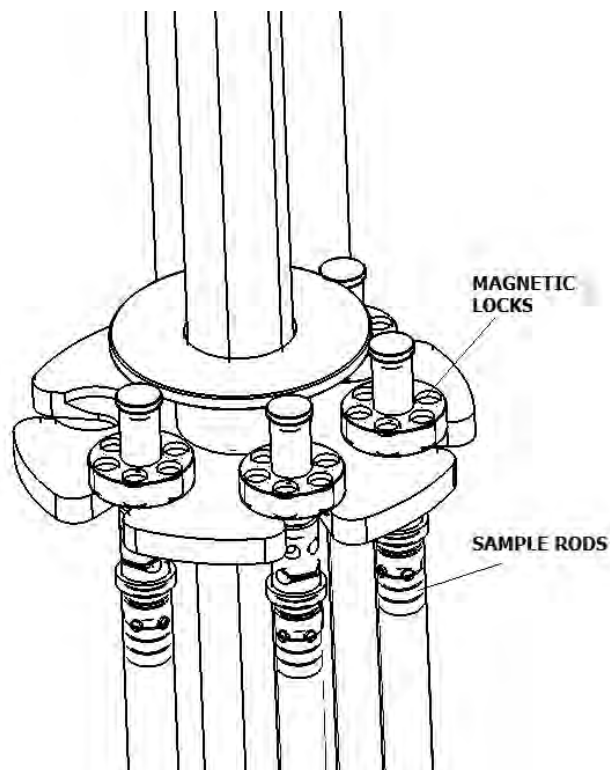


Figure 1-2. Top Detail of the Sample Carousel.

1.2.2 The Sample Rod Vertical Translator and Carriage Arm

The Sample Queue vertical translator and carriage arm rotates, moves, and lifts a selected sample rod to and from the sample chamber. The vertical translator moves the selected sample rods up and down in addition to loading the sample rod onto the sample carousel upper tray.

The carriage arm is equipped with a mechanical “sensing” switch that is used to detect when a sample rod is loaded into the carriage arm. In addition, the sensing switch also detects fault conditions that might prevent a sample rod from being inserted or removed from the VersaLab™ sample chamber.

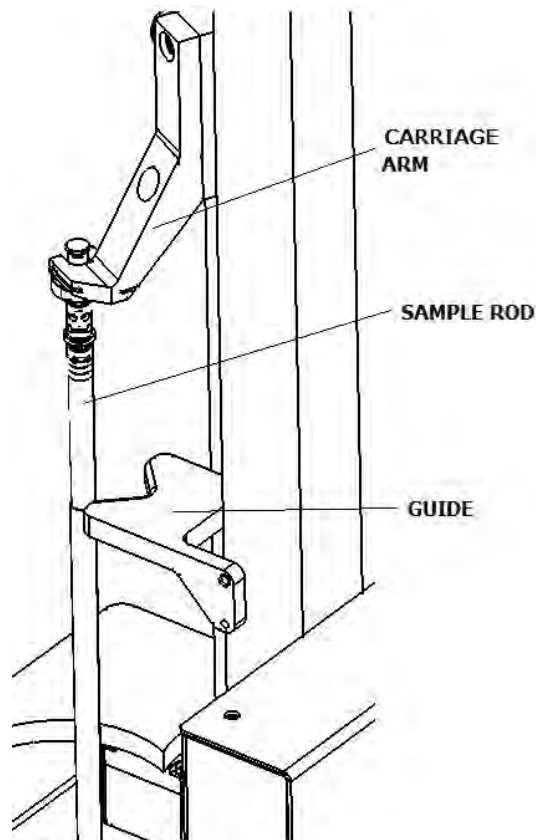


Figure 1-3. Sample translator and carriage arm.

1.2.3 The Sample Chamber Gate Valve Door

Integral to the Sample Queue is a Gate Valve Door which enables the system to automatically seal the VersaLab™ sample chamber. The Gate Valve Door “home” position is when the valve’s door is completely open.

1.2.4 Control Area Network (CAN) Module Electronics

The Sample Queue Control Module electronics (see Figure 1-1) communicate with the VersaLab™ via the Controlled Area Network (CAN) bus. The PC software communicates with the Sample Queue Control Module using CAN over USB via the CANOpen-USB dongle. A CAN module adapter assembly powers the Sample Queue Control Module through the VersaLab™ electronic’s bay just like any other CAN measurement option.

Installing and Removing the Sample Queue Option

2.1 Introduction

This chapter contains the following information:

- Section 2.2 lists the components of the Sample Queue option and describes the procedures you will use for the initial installation on the system.
- Section 2.3 describes the procedures you will use to deactivate and remove the Sample Queue option so that you can use a different measurement option.

2.2 Initial Installation of the Hardware and Software

This section describes the procedures you will use for the initial installation of the VersaLab™ Sample Queue. These procedures apply *only* to the first time you set up and use the Sample Queue option. To re-install the Sample Queue option after it has been deactivated and a different measurement option (e.g., the Heat Capacity option) has been used, you will use the procedures in Section 2.3, "Reconfiguring the system for the Sample Queue Option."

Important: Parts of the initial installation may have been performed at the factory if the Sample Queue option was purchased as part of a new system.

Table 2-1 lists the components of the Quantum Design Sample Queue option. Verify that you have received all the components before you start the installation process.

Table 2-1. Sample Queue system components

COMPONENT	PART NUMBER	ILLUSTRATION
Sample Queue	4300-101	Figures 1-1, 2-1, 2-8, 2-9, 2-13, 3-4
Sample Rods	4096-276	Figures 1-2, 1-3, 2-1, 2-13, 3-1
Sample Queue Drive Cable Assembly	3300-036	Figures 2-9, 3-7
Model CM-R Sample Queue Adapter*	4300-145	Figures 3-8

*This item might be pre-installed.

Installation Process

In the event that you are performing a complete initial installation (i.e., no components were installed at the factory), the process includes the following phases:

- installing and verifying the modular control system, CAN network adapter, and CAN driver software (if no other CAN-based measurement options have been previously installed on your system)
- inserting the control modules
- warming the sample chamber, setting the magnetic field to zero, and venting the sample chamber
- installing the VSM or ACMS II (which ever available) coilset puck
- inserting the sample rod guide tube
- mounting the linear motor transport
- mounting the Sample Queue on top the linear motor transport
- completing the electrical connections
- installing the MultiVu software application and the Sample Queue software
- activating the Sample Queue option
- configuring the Sample Queue coilset

The complete initial installation of the Sample Queue option should take no longer than 30 minutes.

In the event that you are performing a partial installation only, check the instructions for each phase to be sure that you understand critical aspects of the process.

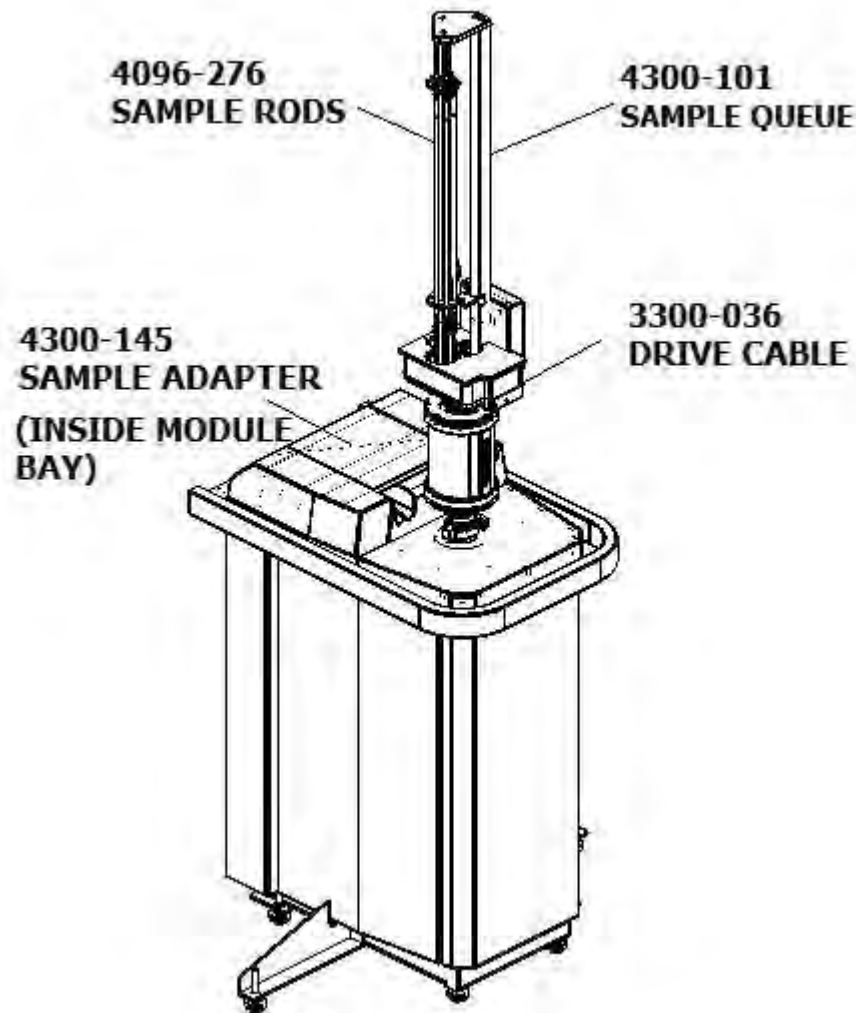



Figure 2-1. System components for the Sample Queue option.

2.2.1 Prepare the System for Option Installation

To prepare the System for installation of the Sample Queue option, you will use the MultiVu application to warm the sample chamber to 300K, set the magnetic field to zero (0) Oe, and vent the sample chamber. Then, you will remove any sample puck or option that is currently installed in the chamber. When you do this, be sure to remove the standard centering ring from the chamber opening (the VSM and ACMS II has a custom-designed centering ring).

1. Set the system temperature to 300 K:
2. Set the field to zero (0) Oe.

	WARNING !
<p>Verify that there are no nearby sources of magnetic field (e.g., NMR or other laboratory magnets) before attempting to install or remove the linear motor transport, as explained in Section 2.2.4 and 2.3.7.</p>	

3. Vent the sample chamber.
4. Remove any sample puck or option that is installed in the sample chamber.
5. Remove the standard centering ring (or any other hardware that is present) from the top flange of the system.

2.2.2 Install the VSM or ACMS II Coilset

The coilset puck contains the VSM or the ACMS II detection coils and a thermometer for monitoring the sample temperature. You will insert the coilset puck into the sample chamber by using the standard puck-insertion tool¹ and the same procedures that are used to insert other types of pucks (see the system User Manual for more information). **Install the puck before you insert the sample tube.**

1. On the system, verify that all items have been removed from the sample-chamber opening, including the standard centering ring.
2. Locate the serial number of the detection coilset puck (4084-850), as shown in Figure 2-2. This serial number will be used to identify the calibration data for the coilset in a later step.
3. Insert the coilset into the sample chamber by using the puck-insertion tool, as illustrated in Figure 2-3.

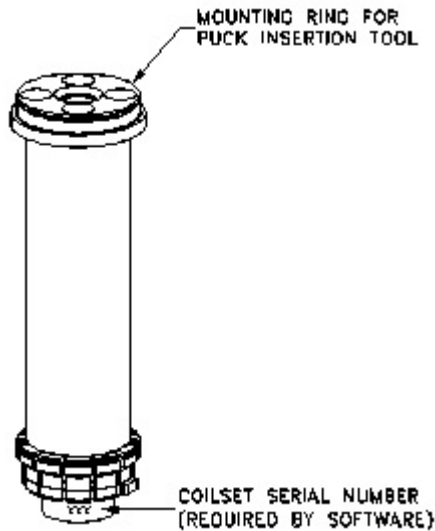


Figure 2-2. Coilset puck, with arrows to coilset serial number and to mounting ring for attaching the puck-insertion tool

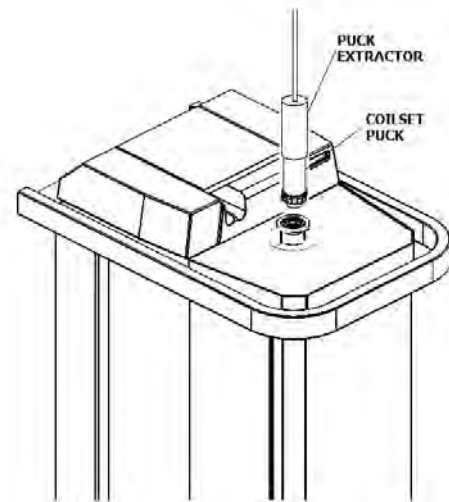


Figure 2-3. Installing the coilset puck with the puck-insertion tool.

¹ The puck-insertion tool is also referred to as the puck-extraction tool, the sample-holder tool, and the sample-insertion (sample-extraction) tool, depending on context.

2.2.3 Insert the Guide Tube

The sample tube contains low-friction bearing sleeves to center the sample rod in the bore of the coilset. Figure 2-4 shows the sample-tube assembly, where you can see that the top of the sample tube assembly includes an integrated centering ring and a stabilizer post. When the sample tube has been inserted into the sample chamber, the stabilizer post will extend into both the sample chamber and the extender tube flange on the sample linear motor transport, as is shown in Figure 2-5. The primary functions of the post are to act as a guide when the transport is installed and to keep the transport on the system.

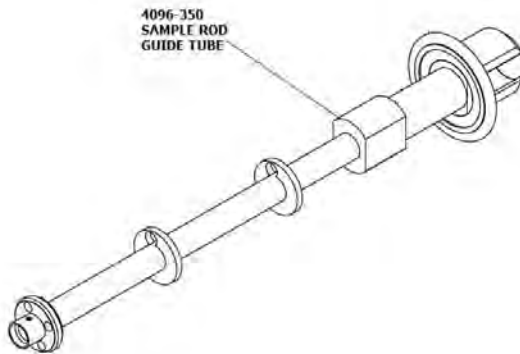


Figure 2-4. Sample rod guide tube assembly (4096-301 is shown for example)

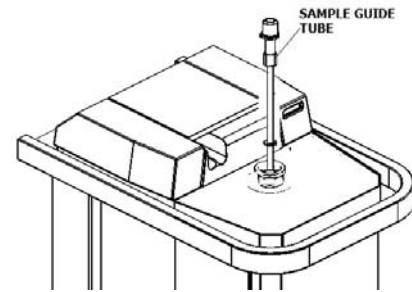


Figure 2-5. Inserting the sample rod guide tube into the sample chamber

Use the steps below to install the guide-tube assembly.

1. Verify that the standard centering ring has been removed from the top of the system. (A VSM and ACMS II-specific centering-ring assembly has been integrated into the sample rod guide-tube assembly.)

Important: You cannot use a standard centering ring between the sample linear motor transport and the sample chamber. As a safety mechanism, the VSM and ACMS II options cannot be installed on the system without the option-specific components, such as the VSM / ACMS II sample rod guide tube assembly with its integrated centering-ring and stabilizer post.

2. Examine the O-ring on the sample-tube centering ring for dust or dirt. If it is dirty, clean it and lightly grease it with silicone vacuum grease.
3. Using Figure 2-5 as a guide, carefully lower the sample tube assembly into the sample chamber until the sample-tube centering ring seats onto the top flange.

2.2.4 Mount the Sample Linear Motor Transport



WARNING !

Verify that there are no nearby sources of magnetic field (e.g., NMR or other laboratory magnets) before attempting to install or remove the linear motor transport, as explained in Section 2.3.7.

The sample linear motor transport (Figure 2-6) moves the sample. You will mount the linear motor transport directly on top of the sample-chamber opening after you have inserted the sample tube into the sample chamber. Before you install the linear motor transport, you must remove the shipping plug and install the extender-tube-flange assembly..

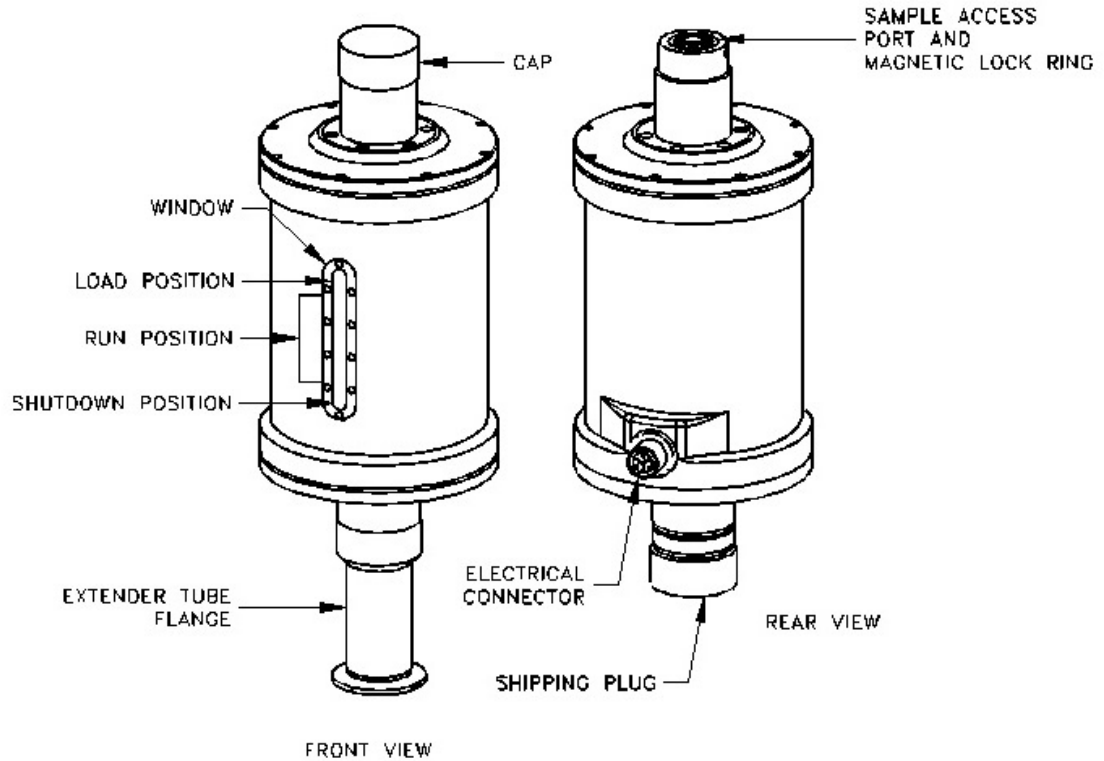


Figure 2-6. Front and rear views of the sample linear motor transport (4096-400). The rear view (right) shows the transport with the shipping plug installed; in the front view (left), the shipping plug has been replaced by the extender tube flange.

CAUTION!

- ◆ Use care when installing the DC linear motor transport—it is fragile, bulky, and moderately heavy (about 10 kg or 22 lb).
- ◆ Always use the VSM or ACMS II-specific parts (e.g., the centering rings) to ensure that the equipment operates safely and properly.
- ◆ Always use the flange clamp (Figure 2-7) to hold the linear motor transport onto the stabilizer post.

1. Prepare the extender tube flange:
 - ☑ Locate the extender tube flange and the flange O-ring (part number VON2-030).
 - ☑ Wipe the neck of the flange with a lint-free cloth (e.g., Kimwipe) to remove any dust or dirt.
 - ☑ Place the O-ring into the neck of the extender tube flange. Firmly press on the O-ring to assure it is completely seated.
 - ☑ Wipe the O-ring and lightly grease it with silicon vacuum grease.

- ☐ Place the extender tube flange on a clean piece of paper or lint-free cloth until it can be installed.
2. Remove the sample linear motor transport and its stand from the storage case and place them on a stable work surface, keeping the motor in a vertical position.
3. Remove the shipping plug from the bottom of the motor (see Figure 2-6).
4. Verify that the O-ring of the extender tube flange is still in place and its exposed surface is clean. If it is dusty or dirty, clean it and lightly grease it with silicon vacuum grease.
5. Screw the extender tube flange onto the bottom of the motor until it is tightly attached, using Figure 2-6 for an example. Hand-tighten the tube **only**.
6. Remove the sample linear motor transport from the stand, keeping it upright. For example, you can support the weight of the linear motor transport by gripping it with one hand on the top tube and the other hand on the extender tube flange.
7. Place the sample linear motor transport onto the top flange of the system by orienting the electrical connector to the rear of the cryostat. The linear motor transport should slide over the stabilizer post at the top of the sample rod guide-tube assembly. Figure 2-7 shows the correct orientation of the linear motor transport, and other relevant parts of the equipment.
8. Verify that the integrated centering ring is sandwiched snugly between the top flange and the linear motor transport.
9. Attach the flange clamp to the flange. **Always use the flange clamp to hold the linear motor transport onto the stabilizer post.**

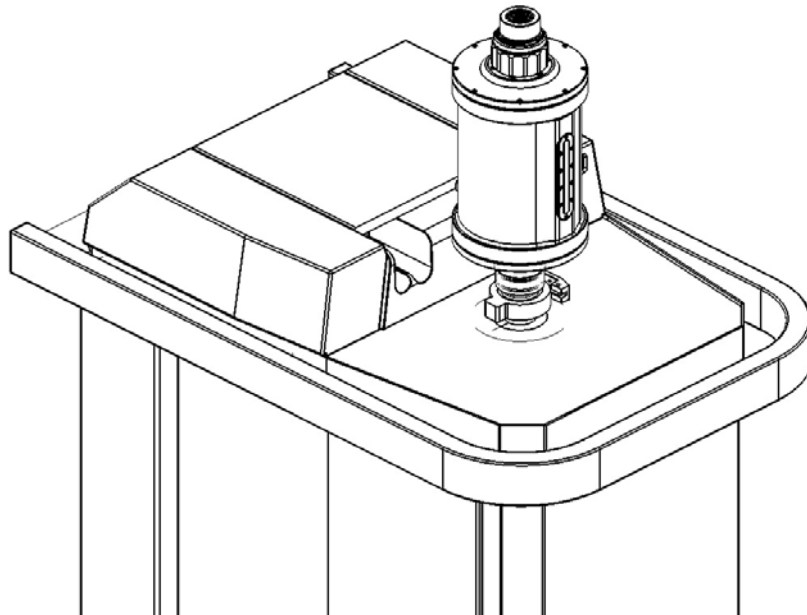


Figure 2-7. Correct orientation of the linear motor on the sample chamber.

2.2.5 Install the Sample Queue Hardware

1. If present, remove the linear transport cap at the top of the sample access port and magnetic lock ring.
2. Make sure the O-ring is clean a lightly greased with silicon vacuum grease.
3. Install the Sample Queue system on top of the sample access port and screw the top linear motor locking nut into the base plate of the Sample Queue systems (see Figure 2-8).

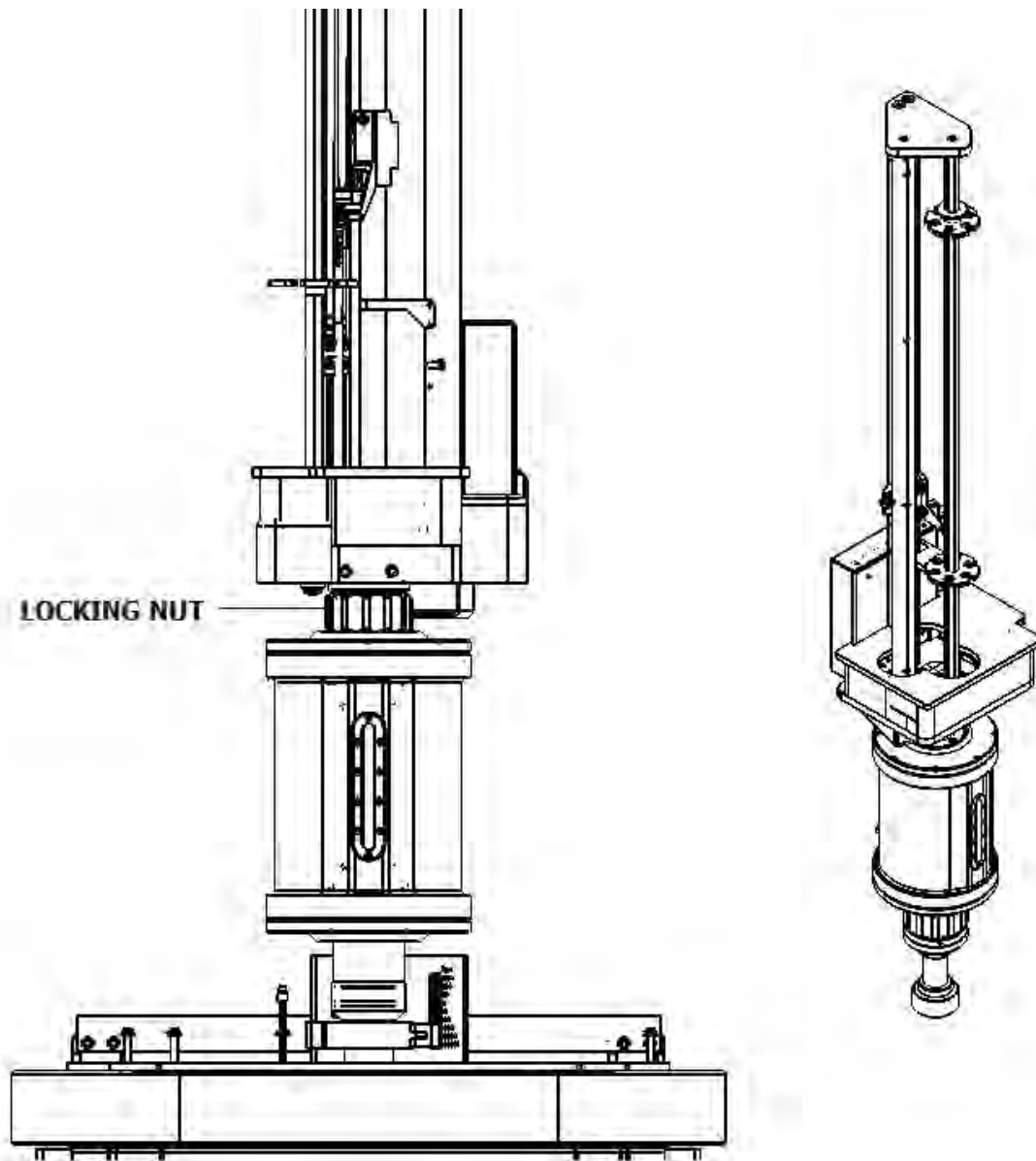


Figure 2-8. Installing the sample linear motor transport on the top flange

2.2.6 Complete the System Connections

Using Figure 2-9 for guidance, complete the electrical connections for the Sample Queue option. After you have attached the connectors, verify that the connections are firm.

1. Install the Sample Queue Module adaptor in the CAN Module Bay. Make sure that the ribbon cable and the adaptor plate are secure.
2. In order to connect the Sample Queue power you will need to shut down the MultiVu application and power down the VersaLab™ in the back of the cabinet.



WARNING !

Verify that the VersaLab™ is powered down before connecting the Drive Cable (3300-036) from the CAM Module adaptor to the Sample Queue Control Module. Failure to do so might result in equipment damage and malfunction.

3. Connect the Drive Cable to the Sample Queue Control Module (depicted in red in Figure 2-9).
4. Connect the other end of the Drive Cable to Sample Changer port JQZ-1 on the adaptor plate.
5. Once the Drive Cable (3300-036) is connected the VersaLab™ is ready to be power up again.
6. Wait for the compressor to re-initialize and then click on MultiVu application icon to re-start the program. Set 300 K.

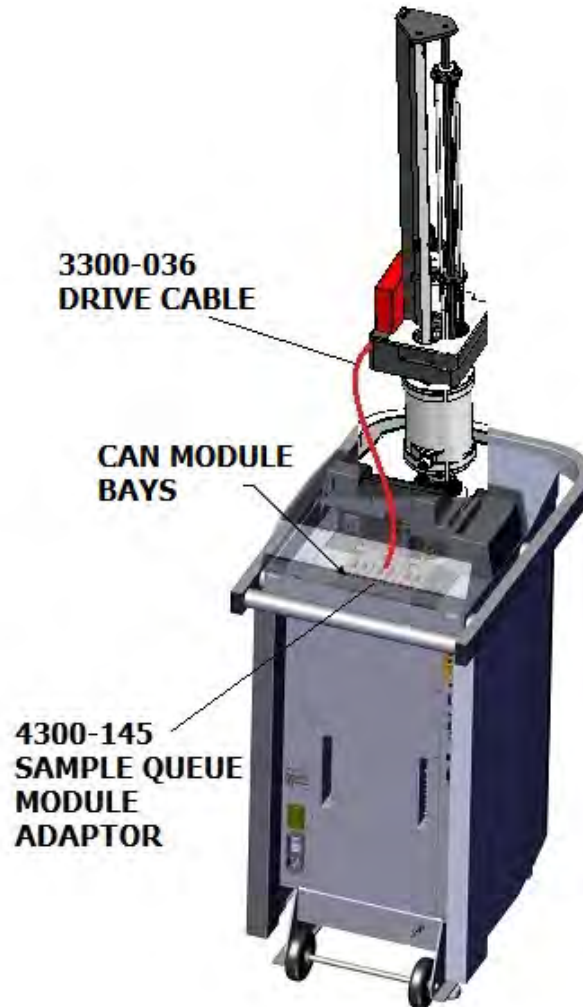


Figure 2-9. Sample Queue option connections on the VersaLab™.

2.2.7 Install the Sample Queue option software

Use the following instructions to install the MultiVu and Sample Queue software applications on your PC. If you purchased the Sample Queue option as part of a new system, you can go to Step 3 below, "Verify that the Sample Queue software is properly installed . . ." See Chapter 4 for more information on the Sample Queue application.

1. Install the most recent version of the MultiVu software if it is not already installed. To install the Sample Queue option software make sure to click on the installer's Sample Queue checkbox.
2. Verify that the Sample Queue software is properly installed by activating it from within MultiVu:
 - a. Start the MultiVu application program.
 - b. Go to the **Utilities** menu on the main MultiVu menu bar (at the top of the application window).
 - c. Select **Utilities >> Activate Option**.

- d. The Option Manager will open. Click on VSM (or ACMS II) under the “Available Options” column and then click the “Activate” button (Figure 2-10).
- e. Once the VSM or ACMS II option is activated then click on Sample Queue and then the “Activate” button.

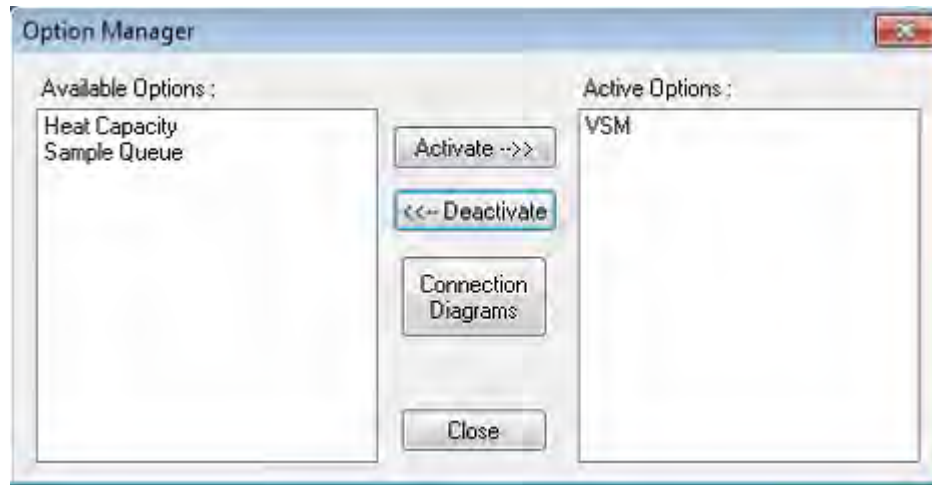


Figure 2-10. Option Manager window

1. As soon as you have activated the Sample Queue option the following will occur:
 - a. The **Sample Queue Control Center** will open (see Figure 2-11). In the control center, you will see a summary of the six sample rods including:
 - o The Sample Queue “**State**” for each of the six sample rods. Note that upon first initialization of the option these States will read “**Empty**”.
 - o Attributes of each of the samples, such as the “**Sample Name/Comments**”, the sample “**Offset**” obtained when mounting the sample in its sample holder.
 - o The Sample Queue’s “**Carousel Slot**” number in which a particular sample holder is mounted into, and the “**Sample Rod**” status which will be either installed in linear motor or not.
 - o Near the bottom of the Sample Queue control center you will see the “**Status**” of the Sample Queue option which upon activation of the option will display “**Ready to Initialize**”.
 - o At the bottom of the Control Center you will see the “**Initialize**”, “**Abort**”, “**Help**” and “**Close**” buttons.
 - o Press the “Initialize” button to initialize the Sample Queue system. An informational window (see Figure 2-12) will now appear asking the user to control that all the moving components of the Sample Queue are not obstructed by any sample rods.
 - o Pressing the “OK” button will start the initialization process. This process consists of a number of processes including: 1) Resetting the module, 2) Homing the carriage arm, 3) moving the carriage arm to the center position, 4) Homing the vertical drive to top of the travel, 5) Homing the carousel, 6) Rotating the carousel slot #1 to the front, 7) Lowering the VSM away from the gate valve door, 8) venting the sample chamber, 9) Homing the door and 10) Closing the door. Once all of these operations are finished the Status will read OK and the Sample Queue is ready to be used.



Figure 2-11. Sample Queue Control Center

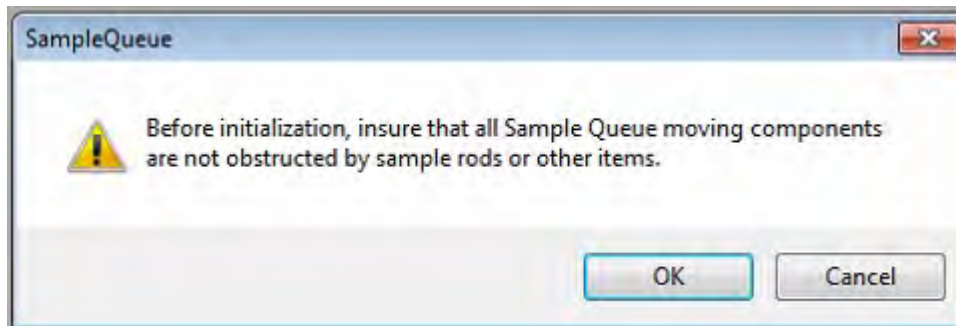


Figure 2-12. Pre-initialization message, click Ok if the path is clear.

2.3 Removing the Sample Queue Option



WARNING!

Verify that there are no nearby sources of magnetic field (e.g., NMR or other laboratory magnets) before attempting to install or remove the linear motor transport, as explained in Section 2.3.7.

You do not need to remove the Sample Queue, the sample linear motor transport and its associated hardware from the system while it is idle. However, if you intend to use it for other types of measurements (e.g., Heat Capacity, Thermal Transport), then you must first remove the Sample Queue and VSM (ACMS II) options.

Summary of Sample Queue and VSM (ACMS II) Removal Procedures

1. Uninstall the sample rod that might be installed in the Linear Motor. The Sample Queue will automatically:
 - a. Set the field to zero.
 - b. Warm up the sample chamber to 300 K, and vent the chamber.
 - c. Open the gate valve door and uninstall the sample rod from the linear motor and load it onto the carousel.
2. Unload all (1-6) of the sample rods loaded into the carousel.
3. Deactivate the Sample Queue option.
4. Close MultiVu and Power down the VersaLab™.
5. Remove the Sample Queue system from the Linear Motor.
6. Prepare for removal of the linear transport and hardware:
 - a. Set the field to zero if it is not already at zero.
 - b. Use the **VSM (or ACMS II)/Remove Sample Wizard** to warm the sample chamber to 300 K, vent the chamber, and move the transport to the load position.
 - c. Shut down the linear motor transport.
7. Deactivate the VSM (or ACMS II) software application.
8. Remove the linear motor transport and place it in the storage case.
9. Remove the sample tube and the coilset puck.

2.3.1 Uninstall the sample rod from the Linear Motor

Open the Sample Queue Control Center (see Figure 2-11) and inspect if any sample rods are installed into the Linear Motor. If no sample rod is installed into the Linear Motor then proceed to the next step. If a sample rod is installed into the Linear Motor then press the corresponding “Uninstall” button. The Sample Queue will then:

- a. Set the field to zero.
- b. Warm up the sample chamber to 300 K, and vent the chamber.
- c. Open the gate valve door and uninstall the sample rod from the linear motor and load it onto the carousel.

2.3.2 Unload all of the sample rods from the Sample Queue carousel

Once all of the sample rods are loaded onto the carousel, these rods need to be removed by the user from the carousel before the Sample Queue system can be removed from the sample linear motor. To do this click one by one the “Unload” button in the Sample Queue Control Center. This will rotate the selected sample rod to the front of the Sample Queue so that it can easily be removed by the user (see Figure 2-13).

CAUTION!

Do not remove sample rods from the carousel unless they are positioned to the front of the Sample Queue system.

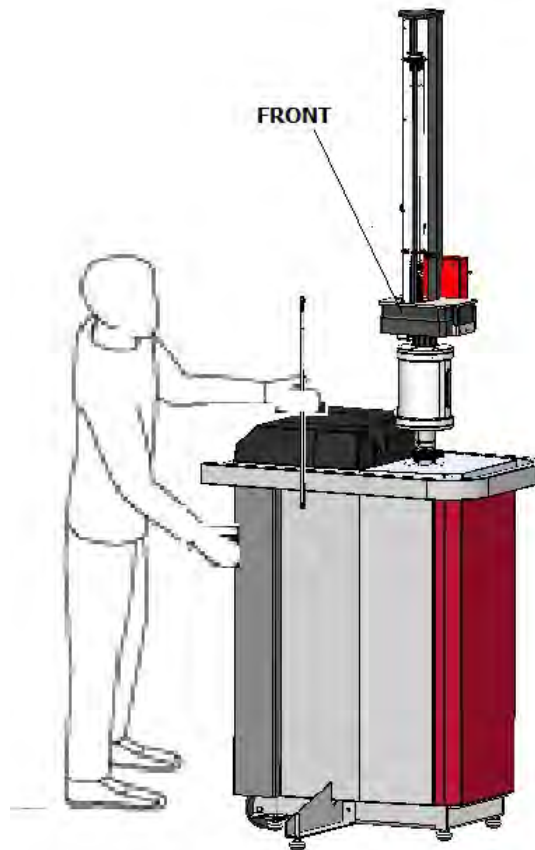


Figure 2-13. Removing a sample rod from the Sample Queue carousel.

2.3.3 Deactivate the Sample Queue option

Once all of the sample rods are unloaded from the Sample Queue carousel the Sample Queue option will need to be deactivated.

1. Select **Utilities >> Activate Option** from the dropdown menu of the MultiVu window (Figure 2-10).


2. When the **Option Manager** dialog opens, select **Sample Queue** and click on the **Deactivate** button. This will move the Sample Queue option from the **Active Options** section of the dialog to the **Available Options** section. The **Sample Queue** window will close, but the MultiVu software application will remain open.

2.3.4 Remove the Sample Queue system

1. Shut down the MultiVu application, and power down the VersaLab™ in the back of the cabinet.
2. Unplug the electrical connector of the Drive cable from the back of the Sample Queue system. (You can leave the other end of the cable connected to the Sample Exchanger Sample Adapter Module.)
Important: Never attempt to move the Sample Queue when it has a cable connected to it.
3. Un-screw the locking nut at the top of the liner motor from the bottom of the Sample Queue system (see Figure 2-8).
4. Slowly lift the Sample Queue until and store it in a safe place.
5. Continue with the VSM or ACMS II removal procedures below.

2.3.5 Remove the Sample Linear Motor Transport

1. Unplug the electrical connector from the back of the sample linear motor transport. (You can leave the other end of the cable connected to the Motor Module.)
Important: Never attempt to move the linear motor transport when it has a cable connected to it.
2. Remove the flange clamp from the top flange of the sample chamber (see Figure 2-7).
3. Slowly lift the linear motor transport until it has cleared the stabilizer post (see Figure 2-7).
4. Place the linear motor transport back in the storage case (4096-150).

	<p style="text-align: center;">W A R N I N G !</p> <p>Store the DC linear motor transport in a secure location to prevent it from being attracted to magnetic fields in the laboratory, including those produced by the superconducting magnet.</p>
---	--

2.3.6 Remove the Sample Rod Guide Tube and VSM (ACMS II) Coilset Puck

1. Remove the sample rod guide tube from the sample chamber.
2. Remove the VSM (ACMS II) coilset puck from the sample chamber by using the puck-extraction tool.²

² See Footnote 1.

3. Unplug the VSM (ACMS II) drive cable from the probe head and set it aside. You do not need to disconnect the other end of the cable from the Motor Module.
4. Return the blank flange to the top of the probe head or install another of the Quantum Design measurement options.
5. Power up the VersaLab™ again.
6. Wait for the compressor to re-initialize and then click on the MultiVu application icon to re-start the program. Set 300K.
7. When the sample chamber has been closed, you can purge and seal it by using the **Chamber** dialog box.
 - ☐ Select **Instrument >> Chamber**.
 - ☐ In the **Chamber** dialog box, click on the **Purge/Seal** button.
6. The base measurement system is now ready for you to install a different option.

Sample Queue Hardware

3.1 Introduction

This chapter contains the following information:

- Section 3.2 describes the basic functionality and hardware components that make up the Sample Queue option.
- Section 3.2 describes the Sample Queue Control Module Electronics.

3.2 Hardware Elements

This section describes the basic hardware components and functions that make up the Sample Queue system. For instructions about installing the various components, please refer to Chapter 2.

3.2.1 Sample Queue Carousel

The Sample Queue carousel function is to store, organize and sequentially rotate up to six sample rods into position for the vertical translator and carriage arm to pick up. The sample carousel is comprised of a post onto which an upper sample rod tray and a lower indexing plate are mounted (see Figure 3-1). The upper tray is mounted approximately 10.6 cm from the top of the post. The indexing plate is mounted 15.2 cm from the bottom of the post. Each rod slot of the sample tray is identified on the carousel by a number on the post.

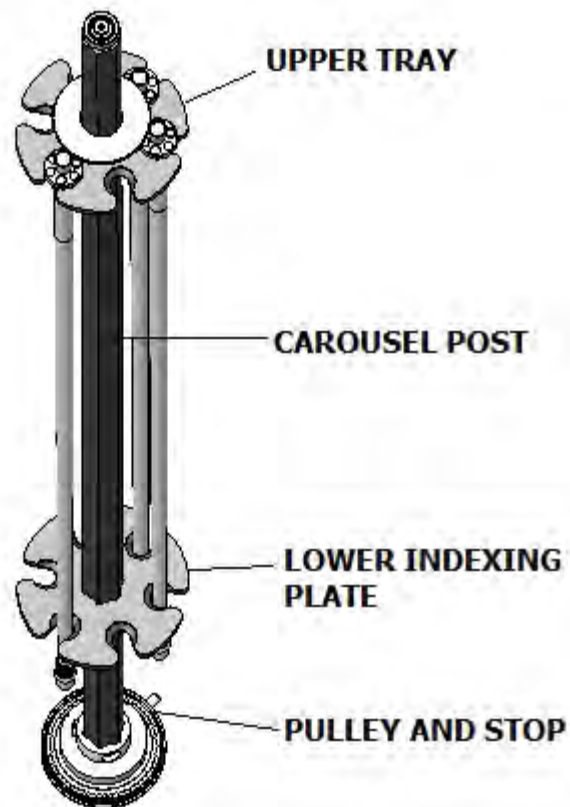



Figure 3-1. Elements of the sample carousel assembly (4300-107).

In the Sample Queue, a 12 Volt stepper motor is used to drive the 315 degrees rotational motion of the carousel. The motor is coupled to a pulley at the bottom of the carousel post via a belt. A subminiature switch is used to mechanically “sense” the position of the carousel and “home” the position of the motor so as to reset its starting position. Using this simple system the user can randomly access, load and unload any sample rods into the sample carousel.

3.2.2 Vertical Translator and Carriage Arm

The Sample Queue vertical translator and carriage arm rotates, picks up and moves the sample rods from the carousel into the linear motor and vice-versa. The translator, the carriage arm and many of its component parts are illustrated in Figure 3-2. The translator post houses the ACME drive which is responsible for the vertical motion of the translator. Two subminiature switches provide for mechanical position limits of the vertical travel.

In general, the sample rod is picked up and held in place in the vertical translator by the carriage arm. A combination of ball bearings on the carriage arm and a magnetic-locking mechanism allow for a precise control of the sample rods in the vertical translator. During a “Load” or “Unload” operation the vertical translator and carriage arm will rotate several degrees during the course of the travel. These rotations are designed to carefully lower the sample rod into the linear motor without hitting edges of the motor and sample chamber interfaces. A 12 Volt stepper motor coupled to vertical translator and carriage arm post pulley via a belt is actuated for this purpose.



WARNING!

Only straight sample rods and sample holders should be used in conjunction with the Sample Queue system. Bent or irregularly shaped sample rods and holders will most likely be caught on edges of the sample transport causing damage to the equipment.

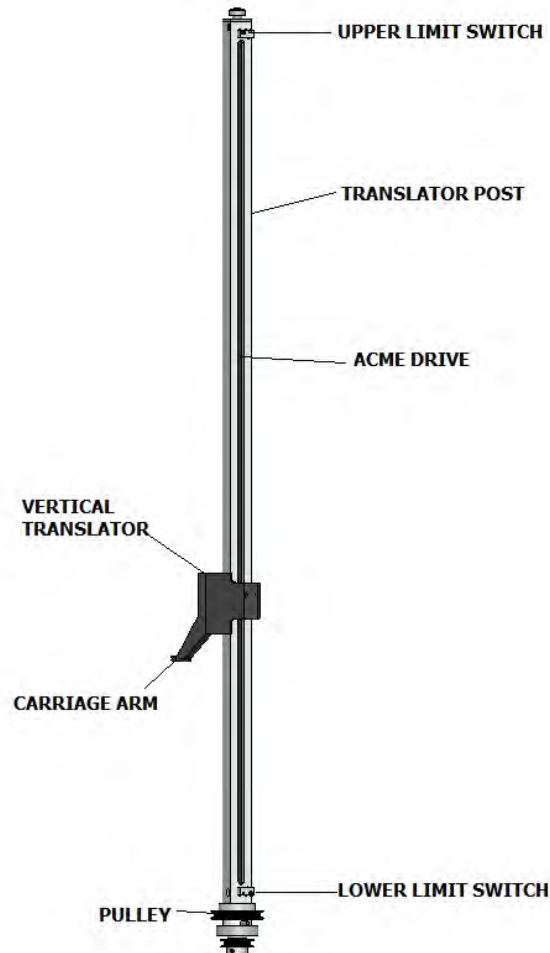


Figure 3-2. Vertical translator and carriage arm assembly (4300-105).

The carriage arm is equipped with a sample rod “sensing” micro switch (see Figure 3-3). The function of this micro switch is twofold: 1) it is used to sense if a sample rod is present on a particular slot of the sample carousel or on top of the linear motor and, 2) sense if the sample rod is stuck on any impediments in its travel. The switch lever (see Figure 3-3) will normally be in a lower vertical position keeping the micro switch open. When the lever moves in the upper vertical position it will close the switch. This will normally mean that a sample rod position has been verified or that a fault condition has occurred.

Important: The “sensing” micro switch should be kept free of dust, debris and dirt. Use a multipurpose duster spray to gently blow away any dirt in the micro switch.

CAUTION!

The micro switch uses an Infra-Red (IR) photo detector transmitter and receiver system to communicate its state (open or closed) to the Sample Queue Control Module. Do not cover or hinder the path directly below the translator arm with any objects as these might prevent the receiver from detecting the state of the switch.

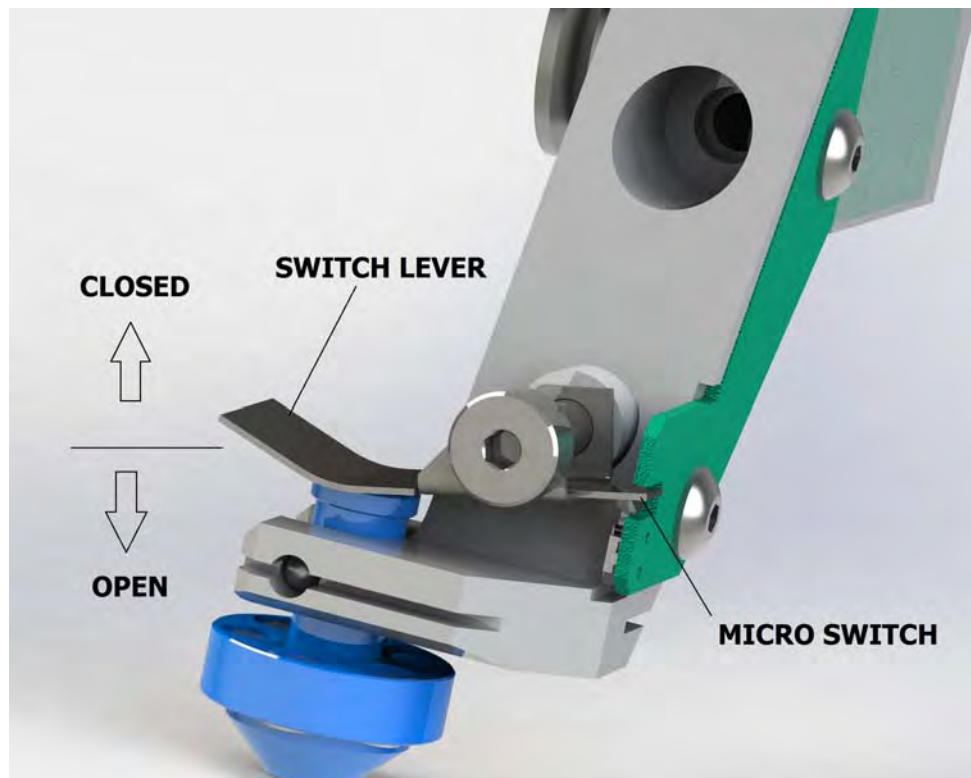


Figure 3-3. Carriage Arm micro switch.

3.2.3 Sample Chamber Gate Valve

The Sample Queue is equipped with a “built-in” sample gate valve which allows the sample chamber to be sealed with respect to atmosphere. Figure 3-4 shows the main elements of the gate valve system: 1) the stepper motor, 2) Subminiature switch and, 3) the gate valve. The switch is used to “home” the position of the valve in the fully open position. A 12 Volt stepper motor actuates the valve and is coupled to the valve pulley using the drive belt.

In Figure 3-4, the flapper guide plate is used to carefully guide the sample rods into the linear motor past the gate valve interface.

CAUTION!

Keep the surfaces of the gate valve clean and free of any debris in order for the valve to make a good seal.

Do not leave any objects in the travel of the gate valve as they will be damaged.

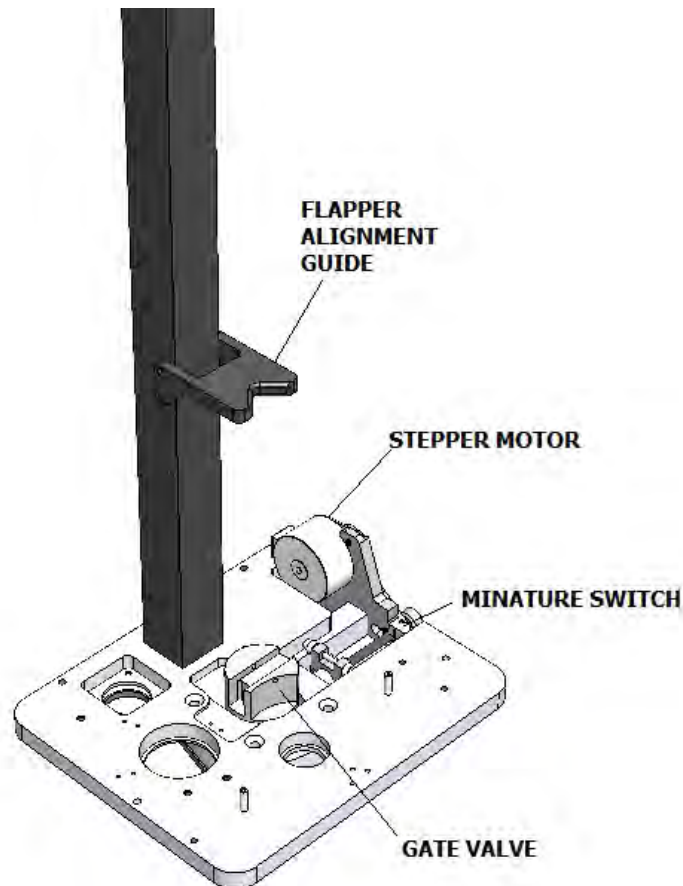


Figure 3-4. Sample Queue gate valve mechanism (not normally in plain view).

3.3 Sample Queue Electronics

This section describes the basic electronic hardware components and functions that make up the Sample Queue system. For instructions about installing the various components, please refer to Chapter 2.

Figure 3-5 describes the general functions of the Sample Queue electrical drive system. The Control Module is powered and it communicates with the VersLab™ via the CAN bus. The microprocessor board in the Control Module is responsible for controlling low level controls of the Sample Queue drive and feedback detection system.

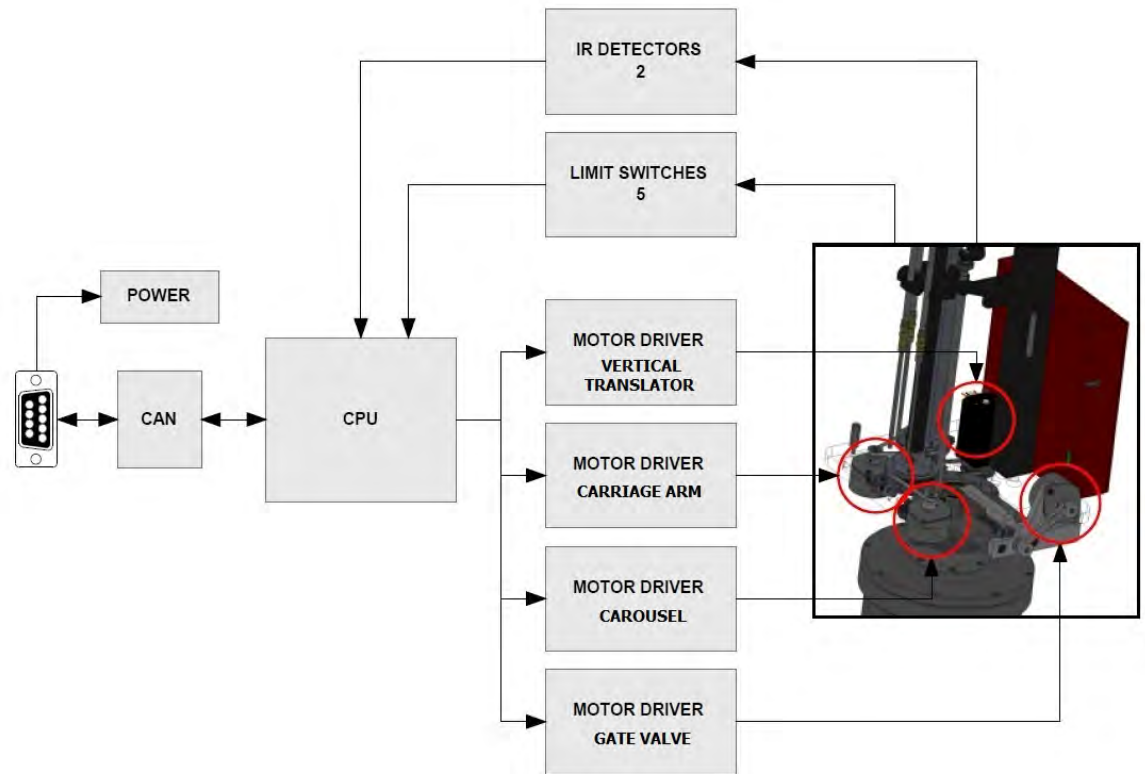


Figure 3-5. Overview of the Sample Queue electronics.

The drive system includes four stepper motor drives for the vertical translator, the carriage arm, the carousel and the gate valve. Five limit switches are used to “home” or reset the travel position of each of these motor drivers. One IR sensor is used for the carriage arm micro-switch to sense a fault condition or verify the location of a sample rod in either the sample carousel or the linear motor. A spare IR sensor is unused at this time.

3.3.1 Control Module

The Sample Queue Control Module is the heart of the Sample Queue drive and feedback detection system. The module is equipped with a microprocessor board (3101-070) that communicates with the Versalab™ via the CAN bus.



Figure 3-6. Sample Queue Control Module

Table 3-1 lists the micro jack connections for the four stepper motor drive circuits. LEDs on the upper right hand corner of control module electronics board indicate proper operation of the system.

Table 3-1. Control Module connections.

MOTOR	MOTOR CABLE (Drive)	SENSOR CABLE (Limit detection)
VERTICAL TRANSLATOR	J4	J5
CARRIAGE ARM	J8	J6
CAROUSEL	J11	J7
GATE VALVE	J12	J9

3.3.2 Drive Cable Assembly

Figure 3-7 shows the Sample Queue drive cable assembly (3300-036), which is the electrical connection between the Control Module and Sample Changer port (JQZ-1) on the Model CM-M adaptor plate in the VersaLab™ electronics bay.

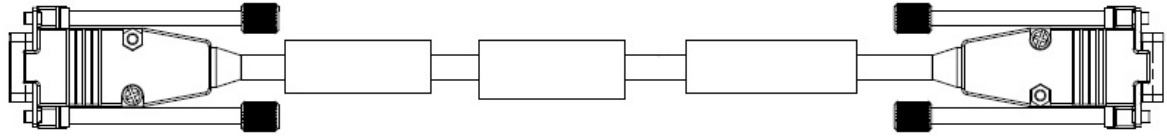



Figure 3-7. Sample Queue signal/drive cable assembly (3300-036).

	WARNING!
<p>Verify that the VersaLab™ is powered down before connecting or disconnecting the Drive Cable (3300-036) from the CAN Module adapter to the Sample Queue Control Module. Failure to do so might result in equipment damage and malfunction.</p>	

3.3.3 Model CM-R Module Adapter Assembly

Figure 3-8 shows the Model CM-R module adapter assembly (4300-145), which provides for a pass through of all the power and logic signals between the VersaLab™ and the Sample Queue control module.

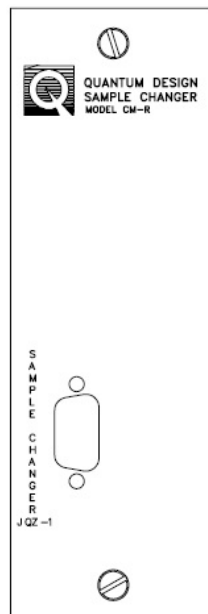


Figure 3-8. Model CM-R Sample Queue module adapter assembly (4300-145).

Sample Queue Software

4.1 Introduction

This chapter contains the following information:

- Section 4.2 describes option overview
- Section 4.3 describes the Sample-Queue specific dialogs and menu items in the MultiVu dropdown menus.
- Section 4.4 describes the **Sample-Queue Control Center** and its components.
- Section 4.5 briefly describes sequence mode commands.
- Section 4.6 describes the Sample Queue data structure.

4.2 Sample Queue Overview

This chapter described the Sample Queue option for the MultiVu application that allows the user to control the operation of the VersaLab™ hardware. The Sample Queue option software combines in a single user interface all the controls to load and install VSM and ACMS II sample rods into the Linear Motor. In addition, Sample Queue sequence commands allow a user to create sophisticated M vs T and M vs H measurement sequences that sequentially measure up to six samples.

Refer to Chapter 2 to learn how to install and activate the Sample Queue MultiVu option.

4.3 Sample Queue Dropdown Menus

Sample Queue-specific actions and commands are all incorporated in the Sample Queue Control Center. For this reason immediate sample operation under the **Sample** pull down menu are no longer available when the Sample Queue option is installed. While this is the biggest difference with MultiVu dropdown menus, the other dropdown menus contain menu items that are common to all Quantum Design systems.

4.3.1 Instrument

The Sample Queue Control center can be accessed from the **Instrument** dropdown menu at the top of the MultiVu window (Figure 4-1). Clicking on the Sample Queue menu item will immediately open the Sample Queue control center.

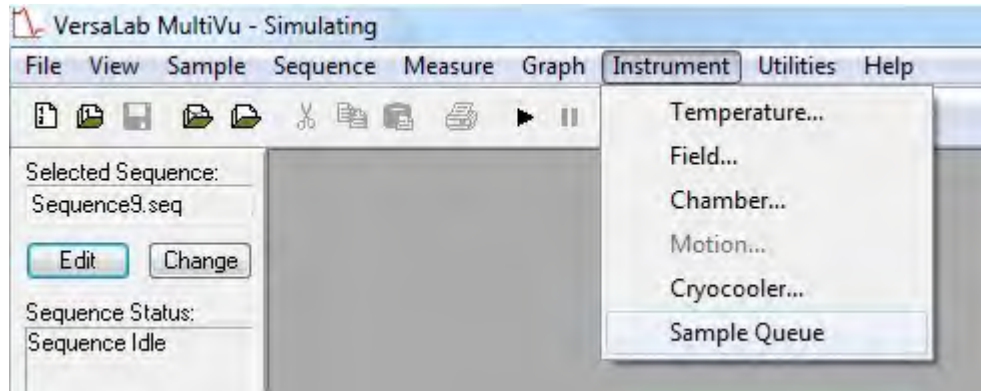


Figure 4-1. The MultiVu window and the Instrument dropdown menu.

4.3.2 Sample

Once the Sample Queue is installed the **Sample** dropdown menu at the top of the MultiVu window will no longer be available as these operations are taken over by the Sample Queue Control Center. If the VSM option is installed then the Sample dropdown menu will look like Figure 4-2.

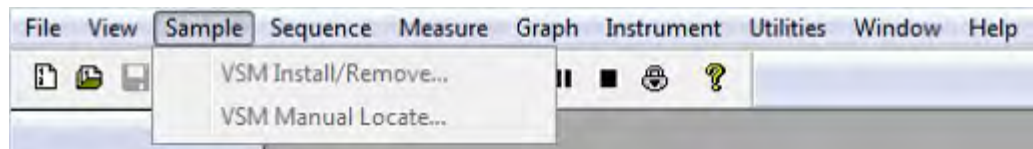


Figure 4-2. The MultiVu window and the Sample dropdown menu showing Sample Queue immediate-mode commands.

4.4 Sample Queue Control Center

The **Sample Queue Control Center** opens within the MultiVu window as soon as you activate the Sample Queue option and application software. Figure 4-3 shows the **Sample Queue Control Center**.

4.4.1 Sample Queue Control Center: Components

4.4.1.1 STATE

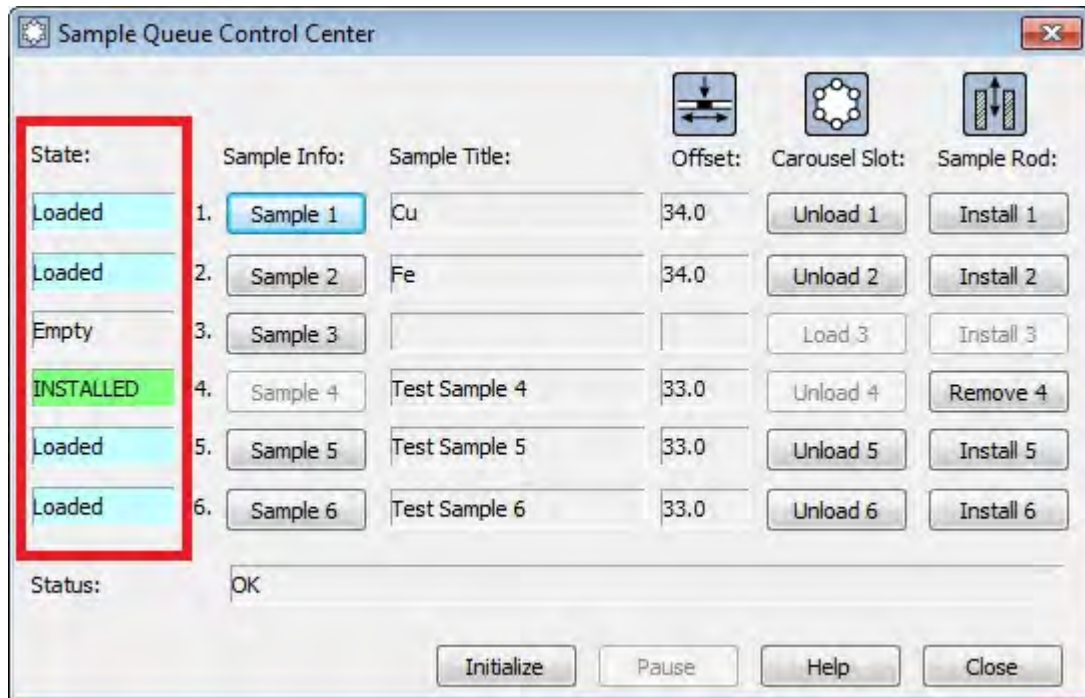



Figure 4-3. State column in the Sample Queue Control Center.

The **State** column is a read only status display of each of the six slots in the Sample Queue carousel. The states are described as follow:

- **Loaded** status indicates that that a sample rod has been loaded into the corresponding carousel slot.
- **Empty** status indicates that that no sample rod has been loaded into the corresponding carousel slot.
- **Installed** status indicates that the sample rod from the corresponding carousel slot has been installed into the Versalab™ sample linear motor ready for VSM or ACMS II measurements.

4.4.1.2 SAMPLE INFO/TITLE

The Sample Info/Title Column (Figure 4-4) is an alpha-numeric user definable field used to label the name of the sample that has been mounted on a particular sample rod. Typically this can be the name of a material, a formula or simply a name of a test.



WARNING !

Do not use punctuation or special characters in the Sample Name/Comments columns as this will interfere with the file structure. Failure to do so might cause the application to crash and a loss of data.



Figure 4-4. Sample Name in the Sample Queue Control Center.

Clicking the Sample location will open the sample information panel for that sample (Refer to figure 4-4a below). The sample properties can be entered into the screen. This information will be recorded in the header of the data file when it is created, and is for information only and is not used in calculating the reported magnetic moment. The offset information is used for the measurement (refer to the following section).

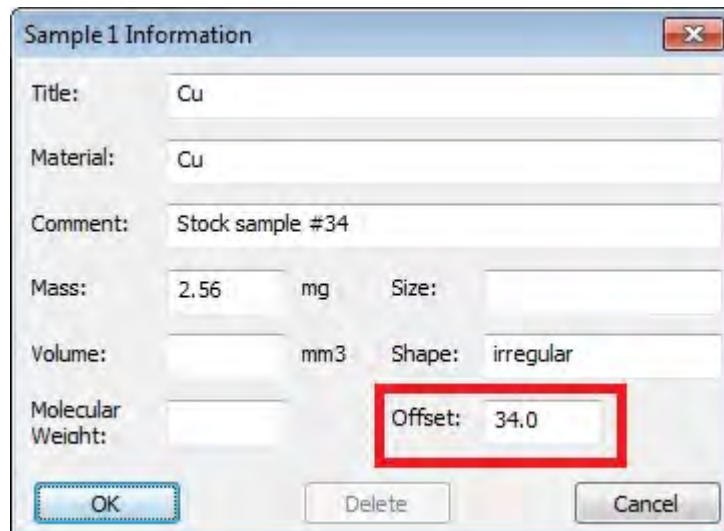


Figure 4-4a. Recording sample properties.

4.4.1.3 OFFSET

The sample **Offset** column (refer to Figure 4-5 left) is a numerical input for the sample mount offset from the Target Line in the sample holder. As described in the VSM and ACMS II User Manuals the sample offset is recorded when first mounting a sample onto a sample holder (Refer to Figure 4-5 right).

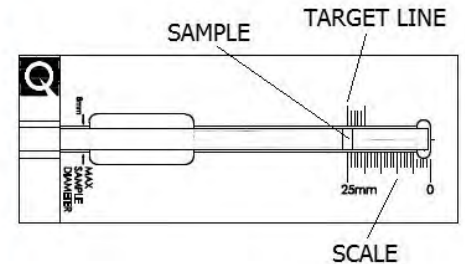
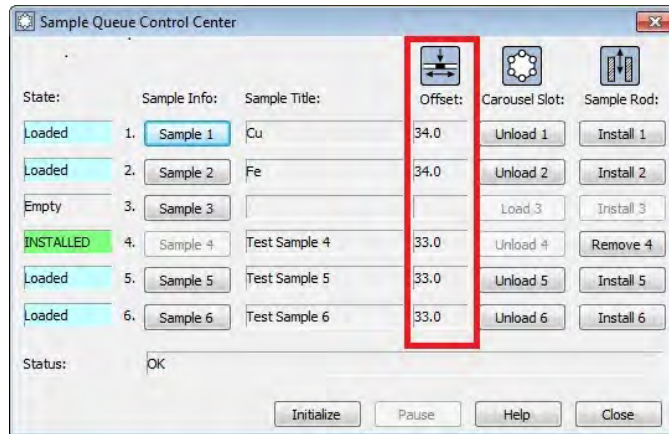


Figure 4-5. Sample Offset in the Sample Queue Control Center.

4.4.1.4 CAROUSEL SLOT: LOAD/UNLOAD BUTTON

The **Carousel Slot** Load/Unload button (Figure 4-6) is used to rotate a particular slot of the sample carousel to the front of the Sample Queue.

- The **Carousel Slot Load** button moves the chosen empty slot to the front of the Sample Queue ready to accept a sample rod from the user.
- The **Carousel Slot Unload** button moves the chosen slot containing a sample rod to the front of the Sample Queue to allow to user to unload the sample rod.



Figure 4-6. Carousel Load/unload button in the Sample Queue Control Center.

4.4.1.5 SAMPLE ROD: INSTALL/REMOVE BUTTON

The **Sample Rod** Install/Remove button (Figure 4-7) is used to install a particular sample rod into the sample linear motor for measurement.

- The **Sample Rod** *Install* button initiates a number of operations which include:
 - Setting the sample chamber temperature to 295K,
 - Venting the sample chamber to atmospheric pressure,
 - Opening the gate valve door,
 - Pick-up the sample rod from the chosen Carousel slot,
 - Lowers the sample rod into the Linear motor,
 - Moves the sample at the bottom of the coilset
 - Closes the gate valve.
- The **Sample Rod** *Remove* button removes the sample rod from the linear transport in a somewhat similar fashion to the Sample Rod Install procedure outlined above.



Figure 4-7. Sample Rod column in the Sample Queue Control Center.

4.4.1.6 STATUS

The **Status** in the **Sample Queue Control Center** dialog box (Figure 4-7), is a read-only display of the system status.

4.4.2 Sample Queue Control Center: "Initialize" Button

The **Initialize** button in the **Sample Queue Control Center** dialog box (Figure 4-7), allows the user to initialize the Sample Queue system upon start up. The initialization process is described in detail in Chapter 2.2.7.

4.4.3 Sample Queue Control Center: "Pause" Button

The **Pause** button in the **Sample Queue Control Center** dialog box (Figure 4-7), might be used in an emergency to stop the current Sample Queue operation. The procedure will stop after the current step of the procedure has been completed.

4.4.4 Sample Queue Control Center: "Help" Button

The **Help** button in the **Sample Queue Control Center** dialog box (Figure 4-7), will open the Online Help for the Sample Queue option. (Not available as of June 2014).

4.4.5 Sample Queue Control Center: "Close" Button

The **Close** button in the **Sample Queue Control Center** dialog box (Figure 4-7) will hide the Sample Queue Control Center window. To re-display it, use the MultiVu menu item Instrument>Sample Queue.

4.5 Overview of Sequence-Mode Sample Queue Commands

Sample Queue sequence-mode commands are, essentially, encapsulated versions of Sample Queue immediate-mode commands.

4.5.1 Sample Queue Control Center: Components

To access these commands the user will need to display the Sequence Command Bar (Figure 4-8) by selecting the “View>Sequence Command Bar” entry while editing a sequence. The Sequence Command Bar contains commands that a user might want to insert into a sequence. In general commands are organized in a tree structure. Click “+” and “-“to expand or collapse tree branches. Double click commands to insert them into the sequence. Sample Queue sequence commands are listed under the “Measurement Commands” branch.

Within the “Measurement Commands” branch a “Sample Queue” sub-branch can be found and expanded by clicking the “+” box next to it. In general, the **Install Sample Rod**, and **Remove Sample Rod** sequence commands (Figure 4-8) found in this branch can be combined with non-Sample Queue sequence commands and looping constructs.

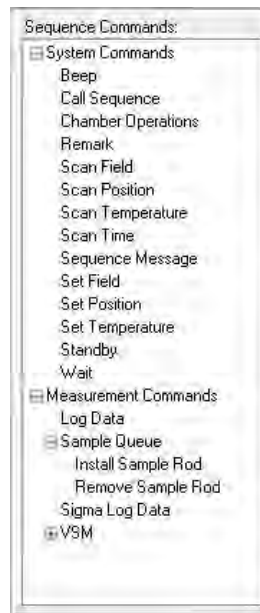


Figure 4-8. Sample Queue Commands in the Sequence Command Control Center.

4.5.2 Sample Rod Install and Remove Commands

To insert a Sample Rod Install operation into a sequence file, click on the **Install Sample Rod** sequence command in the **Sequence Commands** bar. This opens the **Install Sample Rod** Sequence dialog shown in Figure 4-9.

A particular sample rod (number 1 through 6) will need to be entered in the user field. After a number has been entered click the **OK** button to insert the command into the sequence (Figure 4-10).

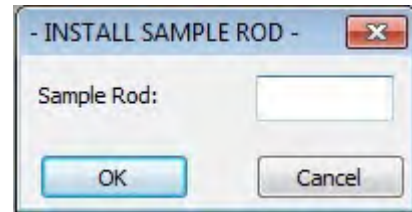


Figure 4-9. Sequence Install Sample Rod popup.

The **Remove Sample Rod** sequence command is similarly inserted into a sequence by simply double clicking this command in the Sequence Command Bar (Figure 4-10).

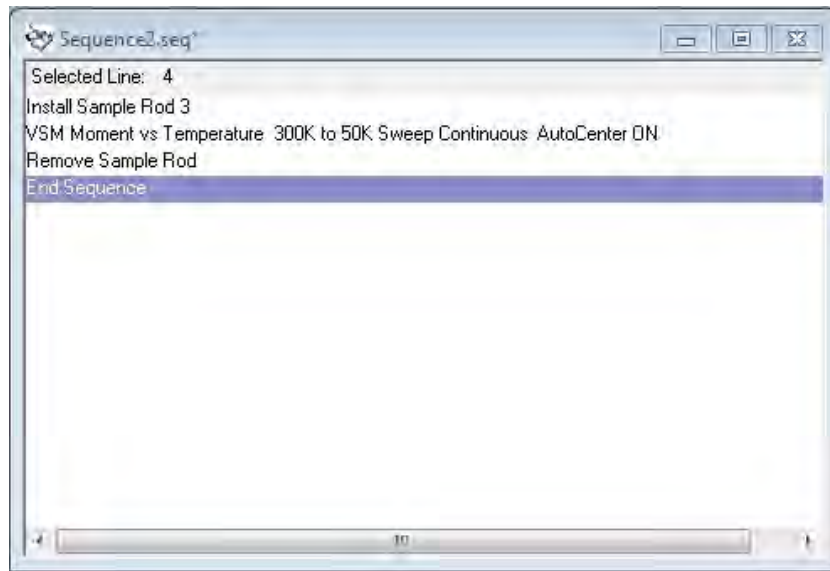


Figure 4-10. Example of Sample Queue commands in the Sequence Editor.

4.5.3 Running a Sequence with missing sample rods

The user can disable sections of a sequence that pertain to sample rods that have not been loaded into the Sample Queue via the Control Center. Figure 4-11 shows a typical sequence written for measuring Moment vs. Field for five sample rods. In this particular instance, however, only sample rod #1 is installed and therefore the user can disable the sections of the sequence that will not be need to be executed.

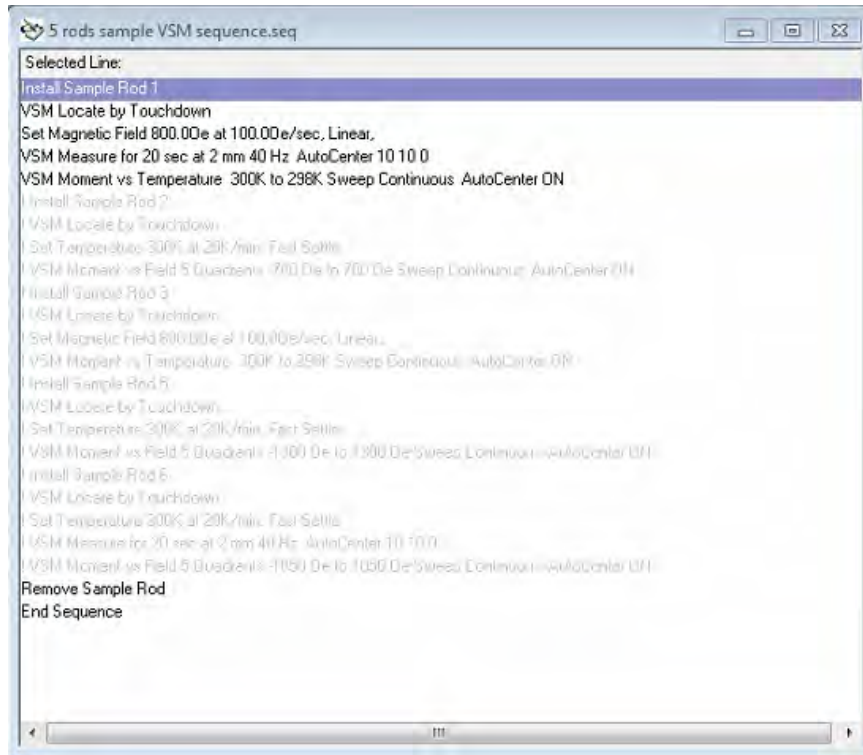


Figure 4-11. Example greyed out commands when the sample rods are not installed in the Sample Queue.

4.6 Overview of the Data File Structure

Because the Sample Queue allows for multiple samples to be measured without user intervention or input during an extended run, the software option allows for the data to be easily associated to a particular sample measured. To this end, the Sample Queue allows the user to either run a sequence with data logged entirely in one data file for all of the six samples (or how many are loaded), or with data logged on separate files as specified in a sequence command for each sample that is measured.

4.6.1 Default setting: Single Data File

As a default, the Sample Queue will log all of the measured data entirely in one single data file. In the raw data view the Sample Queue option will automatically insert a comment every time a sample is switched from one sample rod to another (refer to Figures 4-12 and 4-13). The comment in the data file will reflect the Sample Info/Title and Sample Offset data entered in the Sample Queue Control Center.

Field Name	Field Value
1 Comment	Sample name = Test2, Offset = 32.00 mm
2 Time Stamp (minutes,relative)	27.310
3 Temperature (K)	
4 Magnetic Field (Oe)	
5 Moment (emu)	
6 M. Std. Err. (emu)	
7 Transport Action	
8 Averaging Time (sec)	
9 Frequency (Hz)	
10 Peak Amplitude (mm)	
11 Center Position (mm)	
12 Coil Signal' (mV)	
13 Coil Signal'' (mV)	
14 Range (mV)	
15 M. Quad Signal (emu)	
16 M. Raw' (emu)	
17 M. Raw'' (emu)	
18 Min. Temperature (K)	
19 Max. Temperature (K)	
20 Min. Field (Oe)	

Figure 4-12. Example of Comment inserted in record #245 when a new sample was installed.

Figure 4-13 shows a typical raw view of a typical data file. At the beginning of measurement data bank a comment is inserted (highlighted in grey) to reflect the descriptions that were entered by the user into the Sample Queue Control Center “Sample Name/Comment” and “Offset” columns.

```
[Data]
Comment,Time Stamp (sec),Temperature (K),Magnetic Field (Oe),Moment (emu),M. Std. Err. (emu),Transport Action,Averaging Time (sec),Frequency (Hz),Peak
Sample name = Test1 Offset = 31.00 mm,3609999373.378
"LOOP START",3609999373.46862
Sample name = Test2 Offset = 32.00 mm,3609999530.925
Sample name = Test3 Offset = 33.00 mm,3609999692.681
Sample name = Test5 Offset = 35.00 mm,3609999852.205
Sample name = Test6 Offset = 36.00 mm,3610000009.883
Sample name = Test1 Offset = 31.00 mm,3610000188.337
,3610000240.494,299.777526855469,-0.092254713177681,,2,,,,,17.9900001719594,,,,,299.777526855469,299.777526855469,-0.092254713177681,-0.092254713177681
sample offset = 31 mm touchdown = 8.8900001719594 mm,
3610000290.01,299.797821044922,799.944213867187,-2.20709631561372E-6,3.52109043376275E-6,1,20.0100484152476,40.0298881530762,1.99524087349269,17.980000
,3610000633.6155,299.772232055664,799.963073730469,4.792521753117E-7,1.5673178421176E-5,1,0.999253354069792,40.0298881530762,1.99615574867465,17.980000
,3610000637.511,299.775939941406,799.915954589844,-5.10630247608418E-7,2.64919976818798E-7,1,0.999253354069792,40.0298881530762,1.99580154497557,17.980
,3610000637.7775,299.776092529297,799.911254882813,-3.8288555112912E-7,2.24340721933438E-7,1,0.999253354069792,40.0298881530762,1.99580154497557,17.969
,3610000638.461,299.776168823242,799.913635253906,2.08345796021504E-7,1.69118755346358E-6,1,0.999253354069792,40.0298881530762,1.99610851584782,17.9800
```

Figure 4-13. Example of data raw view in Notepad highlighting the comment automatically inserted at the beginning of a measurement data bank.

4.6.2 Advanced setting: Multiple Data Files

If multiple users are measuring samples within a single measurement run using the Sample Queue, it might be desired to create separate data files for each of the samples/users. This can be easily accomplished by inserting a “VSM New Data File” name command in the sequence before each measurement sub-sequence is initiated. Figure 4-14 shows a typical Multiple Users sequence which specifies a new data file for each sample rod/sample that is measured.

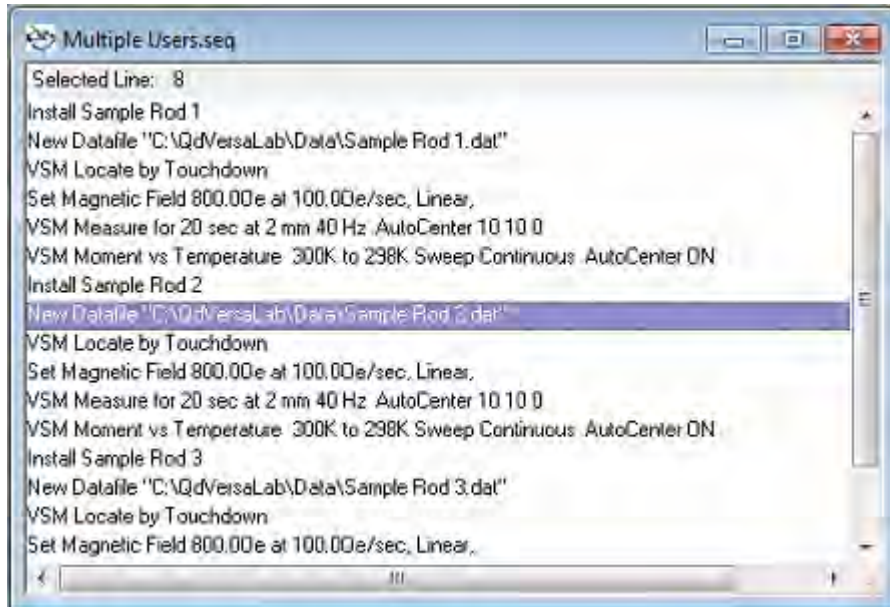


Figure 4-14. Example of Comment inserted in record #245 when a new sample was installed.

4.7 Error Conditions

If the sample rod gets stuck during its movement the “sensing” micro switch will trigger and error and pause the movement of the translation arm. A pop-up window will then appear (Refer to Figure 4-15) which will instruct the user to remove the rod manually and re-initialize the Sample Queue system.

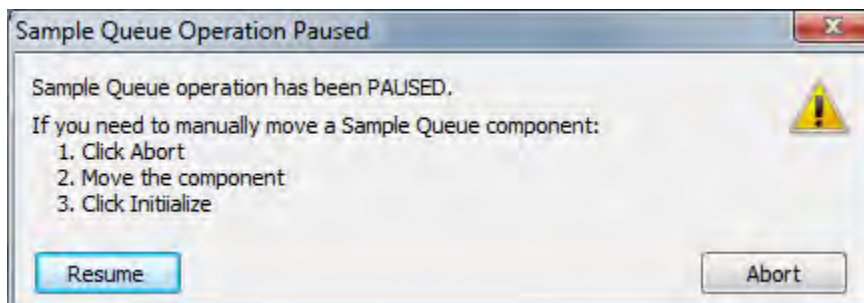


Figure 4-15. Sample Queue Operation Paused pop-up window.

Pin Outs

A.1 Introduction

This appendix contains the following information:

- Section A.3 describes the back panel and relevant components of the Model EM-QZ sample Queue module.
- Section A.3 describes maintenance.

A.1.1 Specifications

Table A-1. Electrical specifications for Sample Queue Control Module

Drive Current Limit	3 A
Supply Voltage	±24 V DC

A.2 Model EM-QZ Sample Queue Module Adapter:

The Control PCB of the module contains an address selector, and the CAN connector through which the module sends and receives network data and receives power.

A.2.1 Address Selector

Each module on the CAN bus must have a unique 5-bit binary address. The selector on the controller board is used to set the four least significant bits, and an internal jumper sets the most significant bit. If the selector is set to zero (0), the module uses its default address. For a Model EM-QZ Sample Queue module, the default address is 24 (or equivalently, “8” on the selector with the internal jumper installed).

A.2.2 QD CAN Connector

The QD CAN connector is the main communication connection for controlling the module. The CAN network signals (CAN High, CAN Low) are connected to all other CAN modules on the bus and to the PC. Power (± 24 volts), reset, and sync signals are also provided to the module through this connector.


Table A-2. QD CAN connector on the rear of the Model CM-A motor module

PIN	FUNCTION
1	-24 V
2	CAN Low
3	Power Return (24V)
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High / Reset
9	+24 V DC

A.3 Maintenance

Wipe and clean the sides of the unit with a clean dry cloth. Do not use lubricants on the Acme screws or belts as this will only attract dirt.

Clean and re-grease the VSM motor top O-Ring regularly, once a month.

	<p>W A R N I N G !</p> <p>As with any CAN module, turn off the power before inserting or removing the Sample Queue power connector.</p>
---	--

Quantum Design is continually working to improve the handling of error conditions by enhancing the software, module firmware, and module hardware. Updates to option software (such as the Sample Queue option), new service notes, and application notes are posted on our website www.qdusa.com. Firmware and hardware updates are handled on an individual basis by Quantum Design service.

If you are encountering performance problems with your motor module after observing the above maintenance steps, please contact your local Quantum Design service representative.



Physical Property Measurement System

AC Measurement System (ACMS II) Option User's Manual

Part Number 1084-800, Rev. A0

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U.S. Patents

5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

Foreign Patents

U.K. 9713380.5 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

C O N T E N T S

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.
- Section P.5 describes the safety guidelines and regulatory information in the manual.
- Section P.6 contains disposal information.
- Section P.7 contains information on how to contact your Quantum Design service representative.

P.2 Scope of the Manual

This manual discusses the Quantum Design AC Measurement System (ACMS II) for the Physical Property Measurement System (PPMS), the PPMS VersaLab™ and DynaCool™ platforms. It contains information about basic functionality, describes the system hardware, and the control software.

The ACMS II measurement option includes a DC Magnetometer measurement mode that can be performed in conjunction with AC susceptibility measurements. This measurement mode is performed with different hardware and software than the stand alone PPMS VSM option. Be sure to read separate VSM measurement option user manuals when making measurements with that option.

P.3 Contents of the Manual








- Chapter 1 provides an overview and theory of the operation of the ACMS II.
- Chapter 2 provides an overview of the ACMS II option installation.
- Chapter 3 describes sample preparation and mounting
- Chapter 4 describes ACMS II measurements types and how to perform them.
- Chapter 5 describes details of the ACMS II hardware.
- Chapter 6 describes details of the ACMS II software.
- Chapter 7 describes common troubleshooting of the ACMS II.
- Appendix A provides a description of the CM-A Motor Module Electronics.
- Appendix B provides a description of the CM-M ACMS II Module Electronics.


P.4 Conventions in the Manual

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

CAUTION!


Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.

	<p style="text-align: center;">WARNING!</p> <p>This symbol signals specific caution or conditions that could result in system damage, bodily harm, or loss of life.</p>
	<p style="text-align: center;">ELECTRIC SHOCK!</p> <p>This symbol signals electrical hazards that could result in bodily harm, or loss of life. Used at all accessible 200-230 V and 380-408 V power outlets.</p>
	<p style="text-align: center;">WARNING!</p> <p>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).</p>
	<p style="text-align: center;">PROTECTIVE CONDUCTOR TERMINAL</p> <p>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.</p>
	<p style="text-align: center;">EUROPEAN UNION CE MARK</p> <p>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.</p>
	<p style="text-align: center;">ALTERNATING VOLTAGE SYMBOL</p> <p>This international symbol indicates an alternating voltage or current.</p>
	<p style="text-align: center;">STANDBY SYMBOL</p> <p>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.</p>



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 users' 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.




This symbol signals information on **fusing**.

P.5 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 ACMS II and reach its best performance.

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse or improper operation of the system.


WARNING!



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 or disconnect the device.

P.5.1 Inspection for Damage

The ACMS II measurement option 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.

	WARNING!
<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.5.2 Electricity


- In case of emergency, switch off the breaker on the back of the pump cabinet labeled “MAIN PWR” and the breaker on the compressor if your system is a cryocooled instrument switch off the main breaker on the compressor.

Observe the following safety guidelines when you use your system:

- 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. In cryocooled instruments, to prevent electrical shock, disconnect the compressor from the power before you install it, adjust it, or service it.
- Keep electrical cords in good working conditions, and replace frayed and damaged 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.
- In general, keep liquids away from the PPMS and ACMS II equipment.
- Keep the PPMS and ACMS II away from radiators and heat sources. Be sure to follow circuit breaker specifications outlined in the PPMS manual.

P.5.3 Magnet Safety

Observe the following precautions for magnet safety. Also make certain you review the material in chapter 1 in the *Physical Property Measurement System: Hardware Manual*.

	WARNING!
<p>Large magnetic fields are dangerous to anyone wearing a pacemaker or other electrical medical device. Make certain any person wearing a pacemaker or similar device stays at least 3.0–4.5 m (10–15 ft.) from the PPMS dewar whenever the PPMS superconducting magnet is charged.</p>	

WARNING!

Large magnets, such as the PPMS superconducting magnets, can attract iron and other ferromagnetic materials with great force. Keep all iron, nickel, and other ferromagnetic objects at least 5.0 m (16.5 ft.) from the PPMS dewar.

The observable effects of magnetic fields are listed in chapter 1 in the *Physical Property Measurement System: Hardware Manual*.

P.5.4 Cryogenic Safety

When you work with cryogenic materials such as liquid helium and liquid nitrogen, keep in mind that (1) they can expand when subjected to room temperature and (2) they can burn.

WARNING!

The extreme cold of liquid and gaseous cryogenics can cause serious burns. Always wear protective clothing, including thermal gloves, eye protection, and covered shoes, when you work with liquid helium, liquid nitrogen, or any other cryogen.

WARNING!

In a poorly ventilated room, helium can displace air and lead to asphyxiation. Always perform cryogen transfers in a well-ventilated room. In the event of a rupture or spill, vent the room immediately and evacuate all personnel.

Cryogenics stored in confined spaces are subject to extremely high pressure buildup, resulting in dangerous explosions. Pressure relief valves are installed on the PPMS dewar and cooling annulus, and rupture disks are installed on the vacuum sleeve and dewar to eliminate the possibility of explosion. *Do not remove, disable, or otherwise tamper with these safety devices.*

P.6 Disposal Information

The ACMS II is excluded from 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 ACMS II 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)

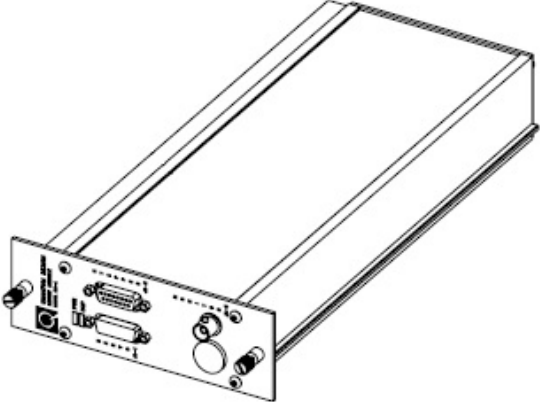
P.6.1 RoHS Statement

The ACMS II is explicitly excluded from the Recast RoHS Directive in Article 2(4)(f) which covers: “*equipment specifically designed solely for the purpose of research and development only made available on a business-to-business basis*”.

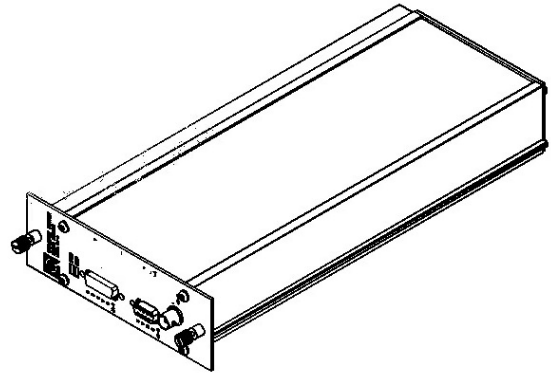
P.6.2 WEEE Statement

WEEE Statement: ACMS II 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.

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

<i>Component Description / Location</i>	<i>Identifying Photograph/Diagram</i>
Motor Module (QD P/N.: 4101-100)	

ACMS II Module (QD P/N.: 4101-430)



P.7 Contacting Quantum Design

If you have trouble with your ACMS II or your system, please contact your local Quantum Design service representative for assistance. See www.qd-international.com for the information about your local representative. You will be asked to describe the problem, the circumstances involved, and the recent history of your system.

Introduction

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the AC measurement system (ACMS II) option.
- Section 1.3 presents the theory of operation.
- Section 1.4 discusses the ACMS II hardware.
- Section 1.5 presents a quick start guide.

1.2 Overview of the ACMS II Option

The Quantum Design AC Measurement System (ACMS II) option for the Physical Property Measurement System (PPMS) is a versatile susceptometer. In addition to a mutual-inductance-based determination of the AC magnetic susceptibility for frequencies between 10 and 10,000 Hz, the coil set permits measurements of the DC magnetization as well, using the DC magnetization (DCM) option, without any change in hardware, sample or sample mount. The state-of-the-art technology of the ACMS II offers extensive susceptibility and magnetization capabilities while retaining a user-friendly interface. By being interfaced with the automated temperature and field control systems of the PPMS, the ACMS II provides a highly powerful, fully automated workstation for the study of magnetic materials.

The ACMS II coil set houses the drive, detection and calibration coils, thermometer, and electrical connections for the ACMS II system. The coil set is used in conjunction with the sample rod guide tube and it fits directly in the PPMS sample chamber. The sample is attached to the end of a rigid sample rod, and it is positioned at the center of the coil set during the measurements. The longitudinal (vertical) motion of the sample is controlled by the sample transport assembly. The linear motor provides rapid and very smooth longitudinal sample motion. The ACMS II sample transport assembly mounts on top of the PPMS probe.

1.3 Theory of Operation

In AC magnetic measurements, a small AC drive magnetic field is superimposed on the external DC field, causing a time-varying magnetization in the sample. The change in flux due to the time-dependent moment induces an emf in the pickup coil, which is proportional to the magnetization, allowing measurements without sample motion. The detection circuitry is configured to detect only in a narrow frequency band, normally at the fundamental frequency (that of the AC drive field), which in the ACMS II is in the range from 10 to 10,000 Hz. On the other hand, the DC magnetization is determined using the DCM option; while the sample is magnetized by the external magnetic field of the PPMS's superconducting magnet, it is made to oscillate around the center of the two detection coils of the coil set. The coil set picks up a signal that is proportional to the magnetization, amplitude and frequency of the vibration.

In order to understand what is measured in AC magnetometry, it is useful to first consider very low frequencies (e.g., 1 Hz), where the measurement is most similar to DC magnetometry. In this case, the magnetic moment of the sample follows the $M(H)$ curve that would be measured in a DC experiment. As long as the AC field is small, the induced AC moment is $M_{AC} = (\partial M / \partial H) \cdot H_{AC} \sin(\omega t)$ where H_{AC} is the amplitude of the driving field, ω is the driving frequency, and $\chi = \partial M / \partial H$ is the slope of the $M(H)$ curve, called the susceptibility. The susceptibility is the quantity of interest in AC magnetometry.

As the applied DC magnetic field is changed, different parts of the $M(H)$ curve are accessed, giving a different susceptibility. One advantage of the AC measurement is already evident: the measurement is very sensitive to small changes in $M(H)$. Since the AC measurement is sensitive to the slope of $M(H)$ and not to the absolute value, small magnetic shifts can be detected even when the absolute moment is large.

At higher frequencies, the AC moment of the sample may not necessarily match the DC magnetization curve, due to dynamic effects in the sample. For this reason, the AC susceptibility is often known as the dynamic susceptibility. In this higher frequency case, the magnetization of the sample may lag behind the drive field, an effect that is detected by the magnetometer circuitry. Thus, the AC magnetic susceptibility measurement yields two quantities: the magnitude of the susceptibility, χ , and the phase shift, φ (relative to the drive signal). Alternately, one can think of the susceptibility as having an in-phase (or real) component χ' and an out-of-phase (or imaginary) component χ'' . The two representations are related by

$$\chi' = \chi \cos \varphi, \quad \chi'' = \chi \sin \varphi \quad (1)$$

$$\chi = (\chi'^2 + \chi''^2)^{1/2}, \quad \varphi = \text{atan2}(\chi'', \chi') \quad (2)$$

where $\text{atan2}()$ is the two-argument $\arctan()$ function.¹

In the limit of low frequency where AC measurement is most similar to a DC measurement, the real component χ' is just the slope of the $M(H)$ curve discussed above. The imaginary component, χ'' , indicates dissipative processes in the sample. In conductive samples, the dissipation is due to eddy currents. Relaxation and irreversibility in spin-glasses give rise to a nonzero χ'' . In ferromagnets, a nonzero imaginary susceptibility can indicate irreversible domain wall movement or absorption due to a permanent moment. Also, both χ' and χ'' are very sensitive to thermodynamic phase changes, and are often used to measure transition temperatures. AC magnetometry allows one to probe all of these interesting phenomena. Typical measurements to access this information are χ vs. temperature, χ vs. driving frequency, χ vs. DC field bias, and χ vs. AC field amplitude

¹ $\text{atan2}(y, x) = \arctan(y/x)$ for $x > 0$; $\text{atan2}(y, x) = \arctan(y/x) + 180^\circ$ for $x < 0$

1.3.1 AC Susceptibility Measurement Process

AC susceptibility measurements do not directly measure a sample's magnetic moment. Rather they measure the derivatives dM/dH . Therefore, samples with very different magnetic moments can still have the same response to a changing magnetic field if their AC susceptibilities are the same in the measurement regimes of interest. The AC susceptibility option in the ACMS II superimposes a small alternating field to the applied external field from the PPMS superconducting magnet, in order to determine the sample's AC magnetic moment response. Both the amplitude and phase of this response are reported. Alternatively, the ACMS II can report the in-phase and quadrature components of the sample's response. Note that the reported amplitude is the amplitude of the *change* in magnetic moment dM and is not an absolute magnetic moment or a susceptibility. To obtain the AC susceptibility, you must divide the amplitude of the change in moment by the amplitude of the alternating field dH . This gives

$$\chi_{ac} = dM/dH. \quad (3)$$

It is important to keep in mind that this is only the local slope of the sample's magnetization curve $M(H)$ and not the "true" susceptibility

$$\chi = M/H. \quad (4)$$

It is important to note the difference between AC susceptibility and "true" susceptibility when dealing with samples that have non-linear magnetization curves.

During an AC susceptibility measurement, an alternating magnetic field generated by the primary of the coil set is superimposed to the DC field of the PPMS superconducting magnet. This alternating field is synthesized by the AC stimulus response (ACSR) high speed electronics' board and sent to a digital-to-analog converter. The analog signal is amplified and used to control the precise AC excitation currents sent to the drive and compensation coils.

By default, the sample undergoes a three-point measurement process that utilizes the calibration coils in order to increase measurement accuracy. The first reading is made with the sample positioned at the center of the bottom detection coil. The sample is then repositioned to the center of the top detection coil and a second reading is taken. A third reading is made with the sample back at the center of the bottom coil. During all three readings, the signals are measured with a lock-in amplifier, conditioned with a low-pass filter, and digitized with an analog-to-digital converter (A/D). The three-point measurement permits comparing the sum of the two bottom coil signals with the top coil signal multiplied by two, such that a correction for drifts of the sample moment and background over the course of the measurement can be made.

When the bottom-top-bottom coil readings are complete, the sample is placed at the bottom of the detection coil. Two more readings are taken with the calibration coil switched into the detection circuit with opposing polarities.

Higher harmonics of the AC susceptibility have been used to probe a number of properties in condensed matter physics, including the strength of the pinning forces in superconducting materials, the peak effect, the distinction between intra- and intergranular superconductivity, the emergence of the pseudo-gap in cuprate superconductors, and spin glass behavior. In its current version (March 2014), the ACMS II is optimized to provide accurate and reliable values for the first harmonic only.

1.3.2 DC Magnetization Measurement Process

The basic principle of operation for the DC magnetization measurement process is that a changing magnetic flux, due to the vibration of the sample concentrically within the coil set, induces a voltage in the pickup coils. This is akin to the operation of a vibrating sample magnetometer. The pickup coil set,

which is shared with the ACMS II, consists of two counterwound coils connected in series and located just above and below the sample, in a first-order gradiometer configuration. The time-dependent induced voltage is given by:

$$V_{coil} = \frac{d\Phi}{dt} = \left(\frac{d\Phi}{dz} \right) \left(\frac{dz}{dt} \right) \quad (5)$$

In equation (5), Φ is the magnetic flux permeating by the pickup coils, z is the vertical position of the sample with respect to the coil, and t is time. For a sinusoidally oscillating sample position, the voltage is based on the following equation:

$$V_{coil} = 2\pi f C m A \sin(2\pi f t) \quad (6)$$

In equation (6), C is a coupling constant, m is the DC magnetic moment of the sample, A and f are the amplitude and frequency of oscillation, respectively.

The acquisition of magnetic moment measurements involves measuring the coefficient of the sinusoidal voltage response from the detection coil. Figure 1-1 illustrates how this is done with the DCM option.

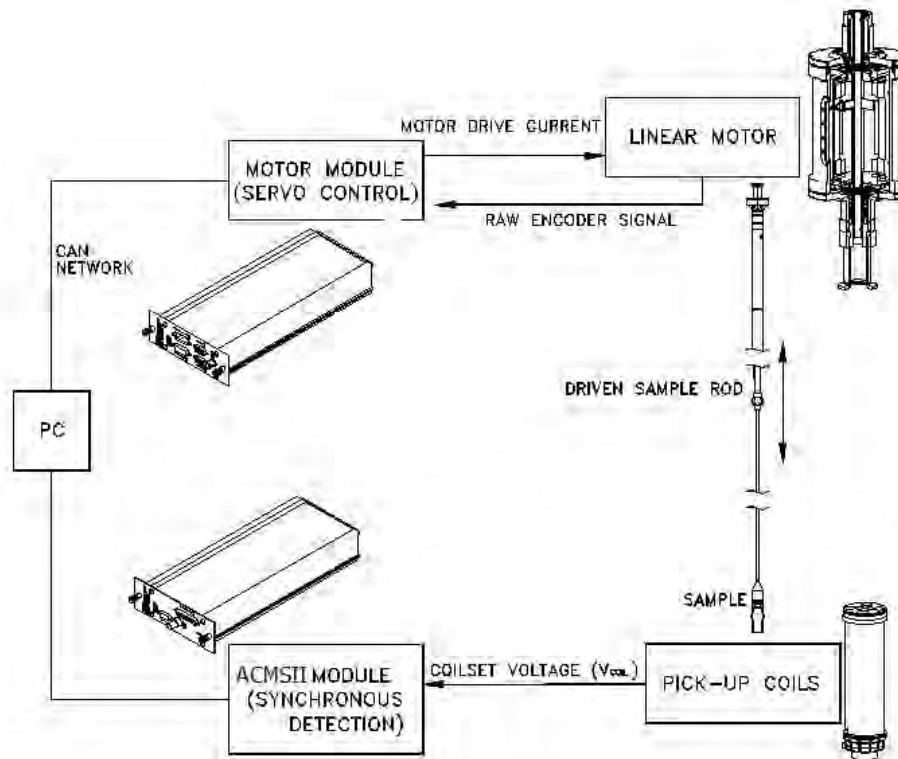


Figure 1-1. Operating Principle for the ACMS II Option.

The sample is attached to the end of a sample rod that is driven sinusoidally. The center of oscillation is positioned at the vertical center of the pickup coil set, which is arranged in a first-order gradiometer configuration. The precise position and amplitude of oscillation is controlled from the DCM motor module using an optical linear encoder signal readback from the DCM linear motor transport. The voltage induced in the pickup coil is amplified and lock-in detected in the DCM detection module. The DCM detection module uses the position encoder signal as a reference for the synchronous detection. This encoder signal is obtained from the DCM motor module, which interprets the raw encoder signals from the DCM linear motor transport. The DCM detection module detects the in-phase and quadrature-phase signals from the encoder and from the amplified voltage from the pickup coil. These signals are averaged and sent over the CAN bus to the MultiVu application running on the PC.

Chapter 5 describes the hardware components of the Quantum Design DCM option in more detail.

1.3.3 Notable Features of the ACMS II System

The Quantum Design ACMS II features a uniquely designed linear motor to move or vibrate the sample. Unlike other vibrating sample magnetometers that use a short-throw resonant voice-coil design, you will find that the DCM linear motor is designed to operate at 40 Hz, with rapid slewing possible over about 6.5 cm of travel.

The sensitivity of the pickup coil is not significantly affected by large magnetic fields, so the DCM can perform sensitive measurements up to the maximum field available from your magnet.

The DCM/ACMS II detection coil is inserted in the sample chamber by using the standard insertion tool assembly. This procedure will make it easy to reconfigure the DCM option with alternate pickup coil designs in the future. You will find that you can change the pickup coil configuration as easily as you can change a puck.

The DCM option is fairly easy to activate or deactivate, just like the other PPMS options. The modularity of the design enables you to perform successive types of measurement with little additional effort. For example, you could follow state-of-the-art DCM measurements with heat-capacity measurements by inserting a different puck or probe.

1.3.4 The ACMS II Coil Set

The ACMS II coil set serves a dual-purpose, to support the measurement of AC susceptibility, using the ACMS II option, as well as the measurement of the DC magnetization, using the DCM option. The detection coils are shared by both options. Refer to figure 1-2.

In support of the AC susceptibility measurements, the coil set contains an AC-drive coil that provides an alternating excitation field, and a detection coil set that inductively responds to the combined sample moment and excitation field. The copper windings for the drive, calibration and detection coils are concentric with each other and with the superconducting DC magnet of the PPMS.

The drive coil is wound longitudinally around the detection coil set. The field amplitude that can be applied depends on the frequency of the alternating field and the temperature within the PPMS probe, but, at any temperature, the drive coil can generate alternating fields of up to 17 Oe in a frequency range from 10 Hz to 10 kHz. As the frequency increases and the temperature decreases, larger amplitude fields can be applied, limited though by self-heating concerns. At temperatures below 25 K, large amplitude fields at sufficiently high frequencies can warm the PPMS sample chamber and ACMS II coil set. See section 4.4.1.2 for more information.

The detection coils are arranged in a first-order gradiometer configuration both to help isolate the sample's signal from uniform background sources, and to provide a reliable centering algorithm for the DC measurements with the DCM. The gradiometer response is optimized for a flat response around the center position, in order to make the magnetic moment insensitive to small variations in the starting position. This configuration utilizes a set of two counterwound copper coils connected in series and separated by about 2 cm. For the DCM measurements, a constant field is applied to the measurement region and the sample is vibrates longitudinally around its position in the center of the gradiometer, inducing a signal according to Faraday's Law. During AC measurements, an alternating field is applied to the measurement region in superposition to the DC field of the magnet, and the sample is positioned in the center of each detection coil. The signal from the detection coils reflects the change in mutual inductance due to changes in the magnetization of the sample.

A compensation coil is situated outside the AC drive coil. The drive coil and compensation coil are counter wound and connected in series so that they receive the same excitation signal. A net field remains within the measurement region, but outside the measurement region the fields from the two coils cancel. It effectively confines the excitation fields to the volume of the coil set, thereby reducing instrument interaction with conductive materials outside the measurement region—that is, the sample chamber walls, the magnet core, and so on—by three orders of magnitude. Each ACMS II system is individually calibrated so that the drive and compensation coils produce the appropriate net excitation signal at the location of the sample.

Each detection coil also contains a low-inductance calibration coil. These two single-turn calibration coils are connected in series and are situated at the center of each detection coil, where the sample measurements occur. During AC measurements, the accuracy of the phase and amplitude calibration is increased by placing the sample between the two detection coils and switching the calibration coils into the detection coil circuit with both possible polarities. The calibration coil is a feature found only on the Quantum Design ACMS II.

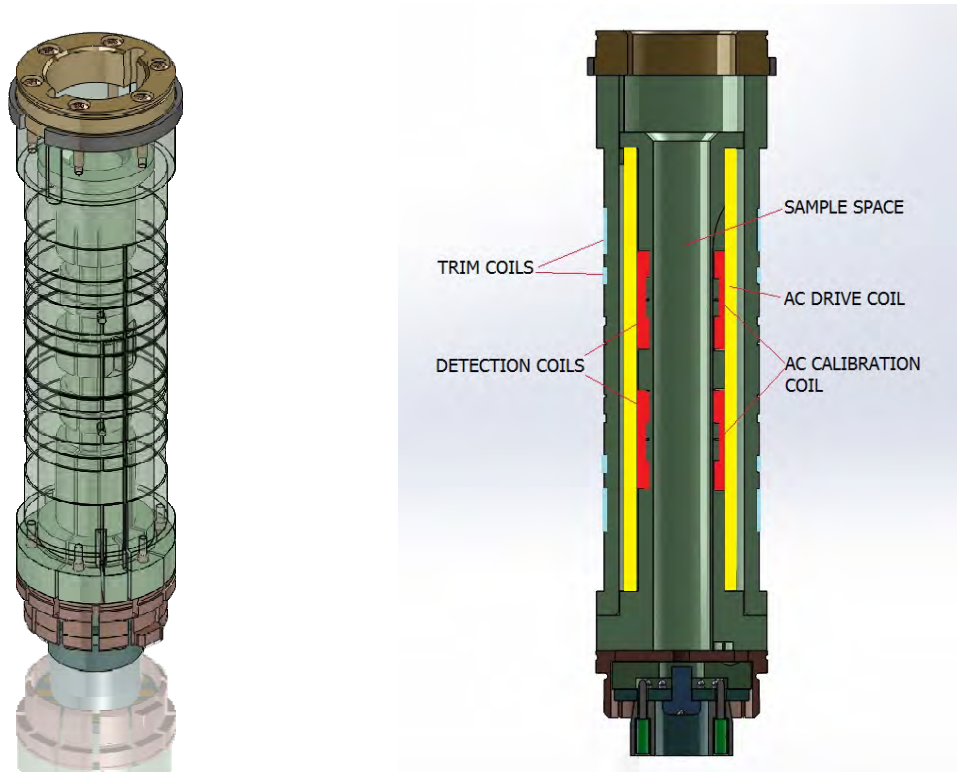


Figure 1-2. DCM and ACMS II Insert and Coil Set

In addition to the alternating field supplied by the ACMS II drive coil, the host PPMS platform can apply a constant field during both AC and DC measurements. The magnitude of the constant field can be up to 16 T, depending on the type of superconducting magnet in the PPMS. The magnetic fields generated by the PPMS superconducting magnets have high homogeneity, of about 0.01% within the measuring region.

One of the most notable features of the Quantum Design ACMS II is its ability to accurately separate real and imaginary components of AC moment response. In light of the resistive and inductive character of the coils, all AC instruments have phase shifts between the drive and the measured signals. These phase shifts are dependent upon parameters such as temperature and AC drive frequency, and therefore susceptible to changes with various measurement parameters. It is important to be able to separate the sample phase shift from this instrumental phase shift. The ACMS II corrects for instrument-dependent phase shift by actually performing measurements in the absence of the sample for each measurement point, using the calibration coils to simulate a sample with an entirely real response as described in section 4.2.

1.3.5 Probe Thermometry

The ACMS II insert is thermally anchored to the base of the PPMS sample chamber, such that the automated temperature control capabilities of the PPMS can be used to control the temperature of the ACMS II sample chamber.

In addition to the PPMS thermometers, the ACMS II has a thermometer mounted directly on its coil form. This thermometer helps reduce errors from thermal lags that may exist, particularly at higher temperatures. The sample maintains close thermal contact with the ACMS II thermometer by virtue of

the thermal conductivity provided by the low-pressure helium vapor in the PPMS sample chamber. This design gives fast thermal response times. The fast thermal response, precise temperature control hardware, and automation software provide accurate, time-efficient, user-friendly thermometry.

1.3.6 ACMS II Control Area Network (CAN) Module Electronics

The ACMS II coils are connected to the PPMS electronics through the 12-pin connector located at the base of the PPMS sample chamber. This configuration uses the existing electronic wiring that is built into the PPMS hardware, thus eliminating the need for additional wiring and connectors.

The ACMS II option includes an ACMS II CAN Module, which is installed in the model 1000 Modular Control System. The ACMS II module provides the electronics that produce the desired excitation current in the drive coils, and it also effectively measures and separates the real and imaginary components of the sample's magnetic response. The heart of the AC board is a high-speed AC stimulus response (ACSR) card that synthesizes the excitation signal and processes the detection coils' response signal.

The ACMS II also uses the DCM Motor module. The Motor module contains the electronics that control the DCM sample transport linear motor.

Installing and Removing the ACMS II Option

2.1 Introduction

This chapter contains the following information:

- Section 2.2 lists the components of the ACMS II option and describes the procedures you will use for the initial installation on the system.
- Section 2.3 describes the procedures you will use to deactivate and remove the ACMS II option so that you can use a different measurement option.

2.2 Initial Installation of the Hardware and Software

This section describes the procedures you will use for the initial installation of the Quantum Design AC Measurement System (ACMS II). These procedures apply *only* to the first time you set up and use the ACMS II option. To re-install the ACMS II option after it has been deactivated and a different measurement option (e.g., the Heat Capacity option) has been used, you will use the procedures in Section 2.2.7, "Activate the ACMS II Option Software."

Important: Parts of the initial installation may have been performed at the factory if the ACMS II option was purchased as part of a new system.

Table 2-1 lists the components of the Quantum Design ACMS II option. Verify that you have received all the components before you start the installation process.

Table 2-1. ACMS II system components

COMPONENT	PART NUMBER	ILLUSTRATION
Linear Motor Transport	4096-400	Figures 2-1, 2-6, 2-7, 2-8, 5-1, 5-2,
Extender tube flange (sometimes referred to as the "Bottom Weldment Flange") and O-rings	4096-418 or 4096-450 and VON2-030	Figures 2-1, 2-6, 2-7, 2-8, 5-1
Storage Case	4096-150	Figure 5-2
ACMS II Coilset Assembly*	4084-850	Figures 2-1, 2-2, 2-3, 4-4, 5-3, 5-4
Sample Guide Tube	4096-301or 4096-350	Figures 2-1, 2-4, 2-5, 5-5
Sample Rods	4096-352or 4096-275	Figures 2-1, 5-6
Sample Holders (paddle-shaped)	4096-392	p. 3-4
Sample Straws	4096-391	p. 3-5
Signal Cable Assembly	3084-801-01 or 3084-801-02 or 3084-801-03	Figures 2-1, 2-8, 5-9
Motor Drive Cable	3096-200 or 3096-201	Figures 2-1, 2-8, 5-10
Model CM-A Motor Module**	4101-100	Figures 2-1, 2-8, 5-11
Model CM-M ACMS II Module	4101-430	Figures 2-1, 2-8, 5-12
ACMS II Option User's Kit	4084-810	Figure 5-7

*This item is shipped in the ACMS II Option User's Kit.

**This item might be pre-installed in the system.

Installation Process

In the event that you are performing a complete initial installation (i.e., no components were installed at the factory), the process includes the following phases:

- installing and verifying the modular control system, CAN network adapter, and CAN driver software (if no other CAN-based measurement options have been previously installed on your system)
- inserting the control modules
- warming the sample chamber, setting the magnetic field to zero, and venting the sample chamber
- installing the ACMS II coilset puck
- inserting the sample rod guide tube
- mounting the linear motor transport
- completing the electrical connections
- installing the MultiVu software application and the ACMS II software
- activating the ACMS II option
- configuring the ACMS II coilset

The complete initial installation of the ACMS II option should take no longer than 30 minutes. In the event that you are performing a partial installation only, check the instructions for each phase to be sure that you understand critical aspects of the process.

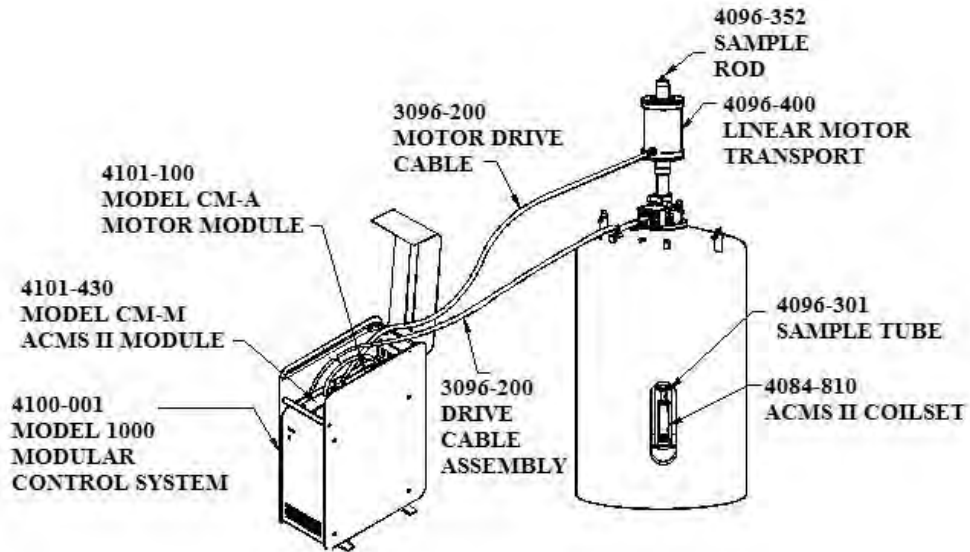


Figure 2-1. System components for the PPMS ACMS II option.

Note: The general setup of the ACMS II option will vary between system platforms.

2.2.1 Install the Modular Control System and CAN Network Adapter (PPMS ONLY)

You will use the instructions in this section only if a Model 1000 modular control system and the driver software have not yet been installed on your PPMS and PC. If your system already has been configured to use Quantum Design modules, you can go to Section 2.2.2.

To install the Model 1000, CAN network adapter, and CAN Manager driver software, refer to the *Model 1000 Modular Control System User's Manual*.

System Verification

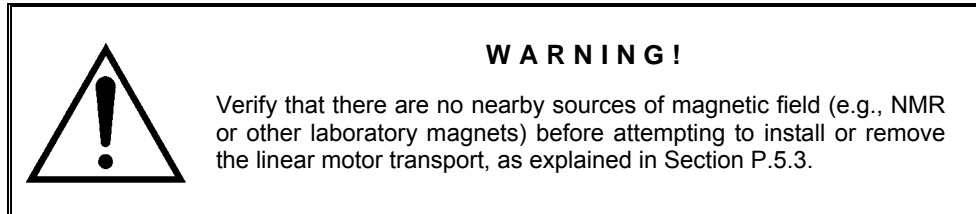
After you have completed installation of the Model 1000 and other components, perform a verification of the system:

1. Verify that the power cable has been connected to the Model 1000 and it has been turned on.
2. Verify that the power LED on the front of the Model 1000 is lit green.
3. Verify that the Model 1000 is connected to the CAN adapter on the PC via the CAN network cable.
- 4.

2.2.2 Prepare the System for Option Installation

To prepare the System for installation of the ACMS II option, you will use the MultiVu application to warm the sample chamber to 300K, set the magnetic field to zero (0) Oe, and vent the sample chamber. Then, you will remove any sample puck or option that is currently installed in the chamber. When you do this, be sure to remove the standard centering ring from the chamber opening (the ACMS II has a custom-designed centering ring).

1. Set the system temperature to 300 K.
2. Set the field to zero (0) Oe.



3. Vent the sample chamber.
4. Remove any sample puck or option that is installed in the sample chamber.
5. Remove the standard centering ring (or any other hardware that is present) from the top flange of the system.

2.2.3 Install the ACMS II Coilset

The coilset puck contains the ACMS II drive and detection coils, and a thermometer for monitoring the sample temperature. You will insert the coilset puck into the sample chamber by using the standard puck-insertion tool¹ and the same procedures that are used to insert other types of pucks (see the system User Manual for more information). **Install the puck before you insert the sample tube.**

1. On the system, verify that all items have been removed from the sample-chamber opening, including the standard centering ring.
2. Locate the serial number of the detection coilset puck (4084-850), as shown in Figure 2-2. This serial number will be used to identify the calibration data for the coilset in a later step.
3. Insert the coilset into the sample chamber by using the puck-insertion tool, as illustrated in Figure 2-3.

¹ The puck-insertion tool is also referred to as the puck-extraction tool, the sample-holder tool, and the sample-insertion (sample-extraction) tool, depending on context.

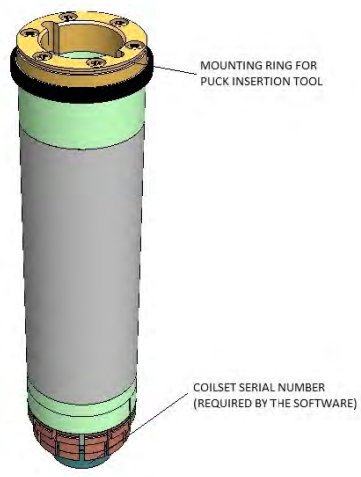


Figure 2-2. Coilset puck (4084-850), with arrows to coilset serial number and to mounting ring for attaching the puck-insertion tool

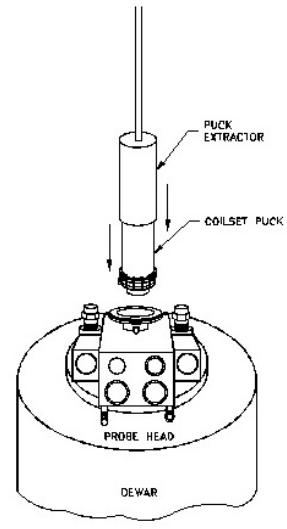


Figure 2-3. Installing the ACMS II coilset puck with the puck-insertion tool.

2.2.4 Insert the Sample Tube

The sample tube contains low-friction bearing sleeves to center the sample rod in the bore of the coilset. Figure 2-4 shows the sample-tube assembly, where you can see that the top of the sample tube assembly includes an integrated centering ring and a stabilizer post. When the sample tube has been inserted into the sample chamber, the stabilizer post will extend into both the sample chamber and the extender tube flange on the sample linear motor transport, as is shown in Figure 2-5. The primary functions of the post are to act as a guide when the transport is installed and to keep the transport on the system. See Chapter 5 for more information on the sample tube.

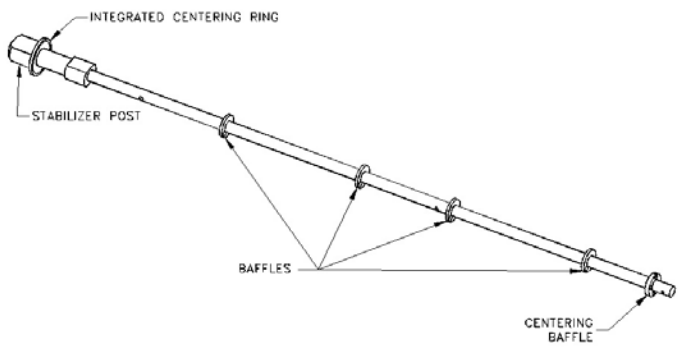


Figure 2-4. Sample rod guide tube assembly (4096-301 is shown for example).

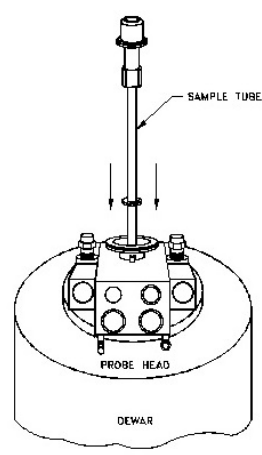


Figure 2-5. Inserting the sample rod guide tube into the sample chamber.


Use the steps below to install the sample-tube assembly.

1. Verify that the standard centering ring has been removed from the top of the system. (An ACMS II-specific centering-ring assembly has been integrated into the sample rod guide-tube assembly.)

Important: You cannot use a standard centering ring between the sample linear motor transport and the sample chamber. As a safety mechanism, the ACMS II system cannot be installed on the system without the ACMS II-specific components, such as the ACMS II sample rod guide tube assembly with its integrated centering-ring and stabilizer post.

2. Examine the O-ring on the sample-tube centering ring for dust or dirt. If it is dirty, clean it and lightly grease it with silicone vacuum grease.
3. Using Figure 2-5 as a guide, carefully lower the sample tube assembly into the sample chamber until the ACMS II centering ring seats onto the top flange.

2.2.5 Mount the Sample Linear Motor Transport



WARNING!

Verify that there are no nearby sources of magnetic field (e.g., NMR or other laboratory magnets) before attempting to install or remove the linear motor transport, as explained in Section P.5.3.

The sample linear motor transport (Figure 2-6) moves the sample. You will mount the linear motor transport directly on top of the sample-chamber opening after you have inserted the sample tube into the sample chamber. Before you install the linear motor transport, you must remove the shipping plug and install the extender-tube-flange assembly. See Chapter 5 for more information on the linear motor transport and its operations.

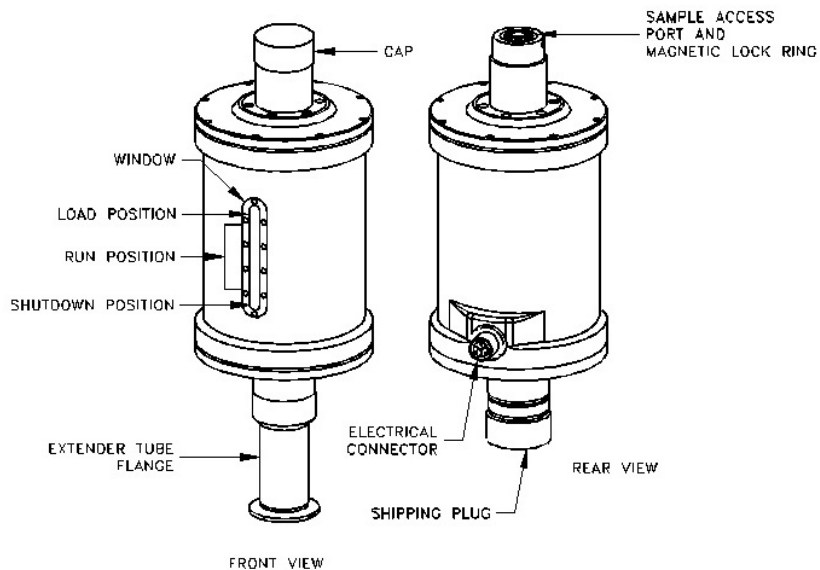


Figure 2-6. Front and rear views of the sample linear motor transport (4096-400). The rear view (right) shows the transport with the shipping plug installed; in the front view (left), the shipping plug has been replaced by the extender tube flange.

CAUTION!

- ◆ Use care when installing the DC linear motor transport—it is fragile, bulky, and moderately heavy (about 10 kg or 22 lb).
- ◆ Always use the ACMS II-specific parts (e.g., the centering rings) to ensure that the equipment operates safely and properly.
- ◆ Always use the flange clamp (Figure 2-7) to hold the linear motor transport onto the stabilizer post.

1. Prepare the extender tube flange:
 - Locate the extender tube flange and the flange O-ring (part number VON2-030).
 - Wipe the neck of the flange with a lint-free cloth (e.g., Kimwipe) to remove any dust or dirt.
 - Place the O-ring into the neck of the extender tube flange. Firmly press on the O-ring to assure it is completely seated.
 - Wipe the O-ring and lightly grease it with silicon vacuum grease.
 - Place the extender tube flange on a clean piece of paper or lint-free cloth until it can be installed.
2. Remove the sample linear motor transport and its stand from the storage case and place them on a stable work surface, keeping the motor in a vertical position.
3. Remove the shipping plug from the bottom of the motor (see Figure 2-6).
4. Verify that the O-ring of the extender tube flange is still in place and its exposed surface is clean. If it is dusty or dirty, clean it and lightly grease it with silicon vacuum grease.
5. Screw the extender tube flange onto the bottom of the motor until it is tightly attached, using Figure 2-6 for an example. Hand-tighten the tube **only**.
6. Remove the sample linear motor transport from the stand, keeping it upright. For example, you can support the weight of the linear motor transport by gripping it with one hand on the top tube and the other hand on the extender tube flange.
7. Place the sample linear motor transport onto the top flange of the system by orienting the electrical connector to the rear of the cryostat. The linear motor transport should slide over the stabilizer post at the top of the sample rod guide-tube assembly. Figure 2-7 shows where the stabilizer post emerges from the sample chamber, the correct orientation of the linear motor transport, and other relevant parts of the equipment.
8. Verify that the integrated centering ring is sandwiched snugly between the top flange and the linear motor transport.
9. Attach the flange clamp to the flange. **Always use the flange clamp to hold the linear motor transport onto the stabilizer post.**

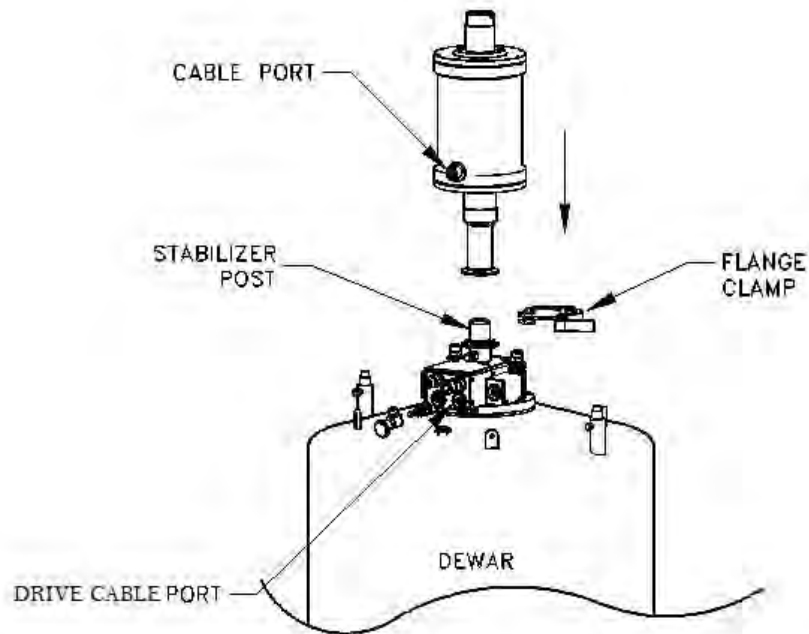


Figure 2-7. Installing the sample linear motor transport on the top flange.

2.2.6 Complete the System Connections

Using Figure 2-8 for guidance, complete the electrical connections for the ACMS II option. After you have attached the connectors, verify that the connections are firm.

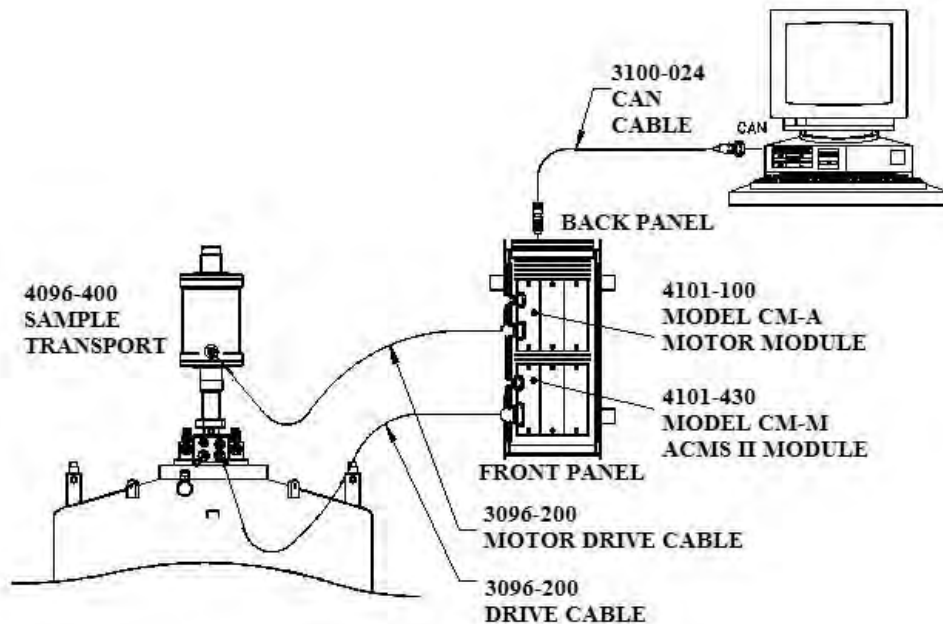


Figure 2-8. ACMS II option connections on the PPMS.

2.2.7 Activate the ACMS II Option Software

Use the following instructions to install the MultiVu and ACMS II software applications on your PC. If you purchased the ACMS II option as part of a new system, you can go to Step 3 below, "Verify that the ACMS II software is properly installed . . ." See Chapter 6 for more information on the ACMS II application.

1. Install the most recent version of the MultiVu software if it is not already installed.
2. Install the ACMS II software by starting the ACMS II software setup wizard and following the instructions.
3. Verify that the ACMS II software is properly installed by activating it from within MultiVu:
 - a. Start the MultiVu application program.
 - b. Go to the **Utilities** menu on the main MultiVu menu bar (at the top of the application window).
 - c. Select **Utilities >> Activate Option**.
 - d. The Option Manager will open. Click on the ACMS II and then on the "Activate" button (Figure 2-9).

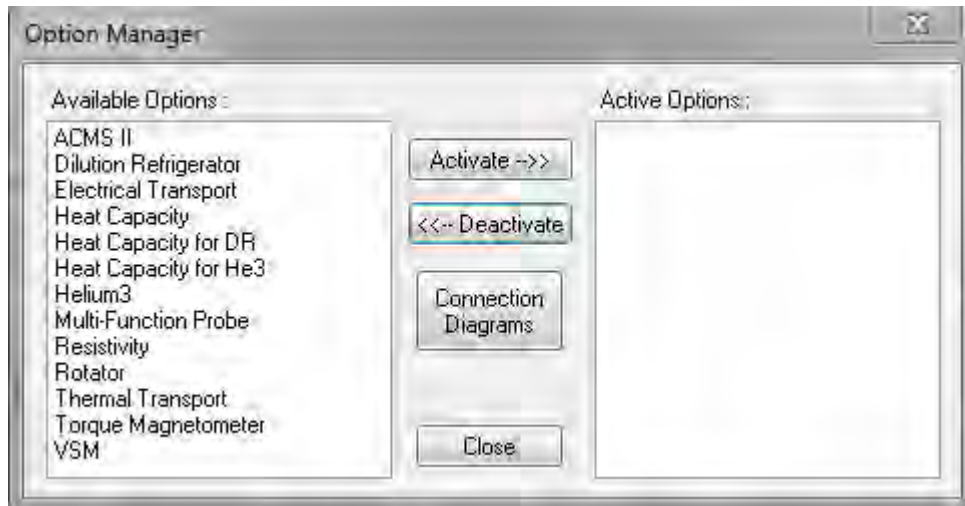


Figure 2-9. Option Manager Window.

5. Three events will occur as soon as you have activated the ACMS II option.
 - The **ACMS II Log** window and the **ACMS II Control Center** will open (see Figure 2-10). In the control center you will see four panels or "tabs": **Install**, **Data File**, **Sample**, and **Advanced**. The **Install** tab is usually at the front when the control center opens. (When running in simulation mode, the **ACMS II Control Center** is titled **ACMS II**.)

Note the **Configure ACMS II System** button under the **Chamber Status** area of the **Install** panel; you will use this button to verify and test the coilset calibration in the next phase.
 - The sample linear motor transport will perform a **Home** (or homing) operation. During a homing operation the system finds the full range of travel for the transport by going through a full travel cycle. The cycle ends at the top in the sample-load position.
 - The **View**, **Sample**, and **Measure** menus on the MultiVu menu bar will show ACMS II -specific features. For example, the **Status** bar at the bottom of the **ACMS II**

Control Center (Figure 2-10) reads "ACMS II Ready," and ACMS II-specific commands are accessible on the **Measure** dropdown menu (Figure 2-11). See Chapter 6 for a full description of the ACMS II software.

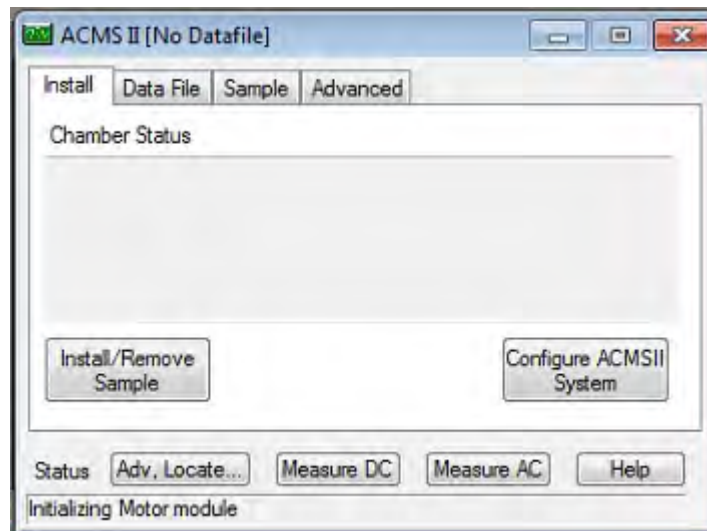
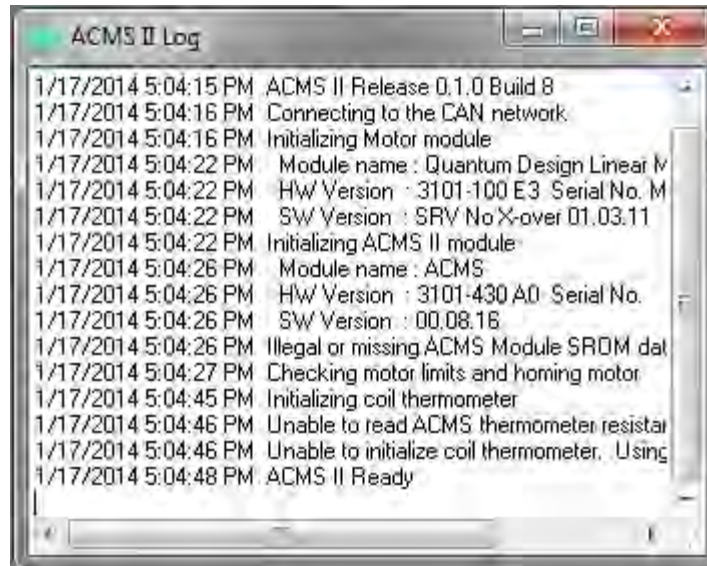


Figure 2-10. ACMS II Log (top) and Control Center (bottom).

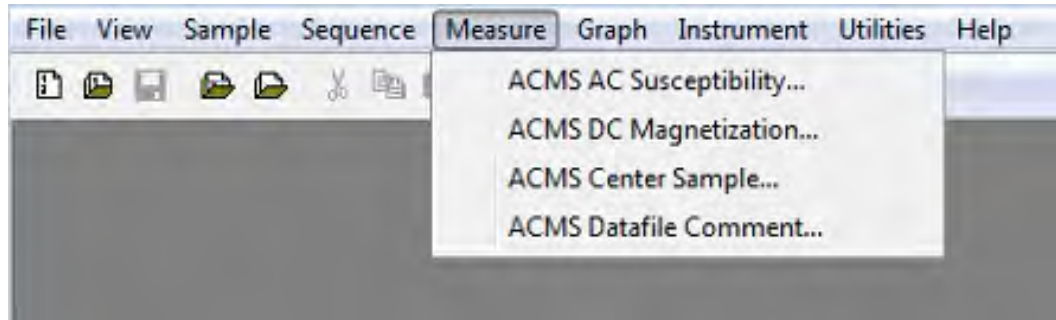


Figure 2-11. MultiVu menu bar and Measure dropdown menu for ACMS II option.

5. Next, you will verify the serial number on the detection coilset puck and test the coilset thermometer and system calibration.

2.2.8 Configure the ACMS II System

The serial number of the detection coilset puck identifies the calibration file that is used to calibrate the coilset. You must verify that the serial number on the puck, which you obtained in Section 2.2.3, matches the standard calibration file referenced in the software. You will use the **Configure ACMS II System** dialogs to specify the puck serial number and test the hardware.

1. Locate the puck serial number that you obtained in Section 2.2.3.
2. Click on the **Configure ACMS II System** button, which is located on the right-hand side of the **Install** tab (Figure 2-10).
3. Page 1 of the **Configure ACMS II System** dialog will open (Figure 2-12). Note the text box where you will enter the serial number of the calibration file.

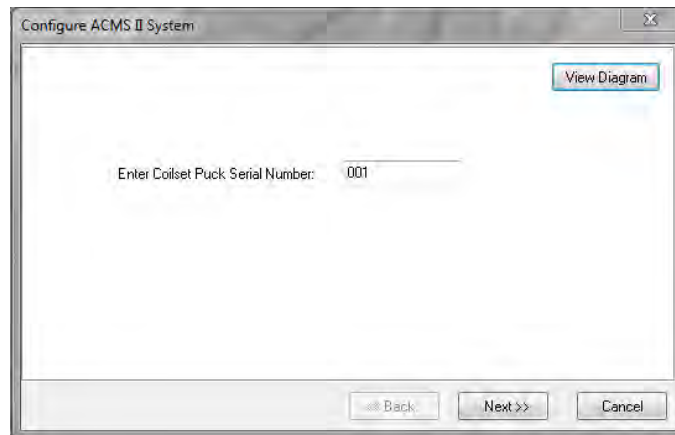


Figure 2-12. Configure ACMS II System dialog, page 1.

4. Verify that the number in the text box matches the serial number of your detection coilset puck. If no number is displayed, enter the serial number of your detection coilset puck in the text box.
5. Click on the **Next >>** button at the bottom of the dialog box. Page 2 of the **Configure ACMS II System** dialog (Figure 2-13) will open, showing the results of system tests to verify system operations.

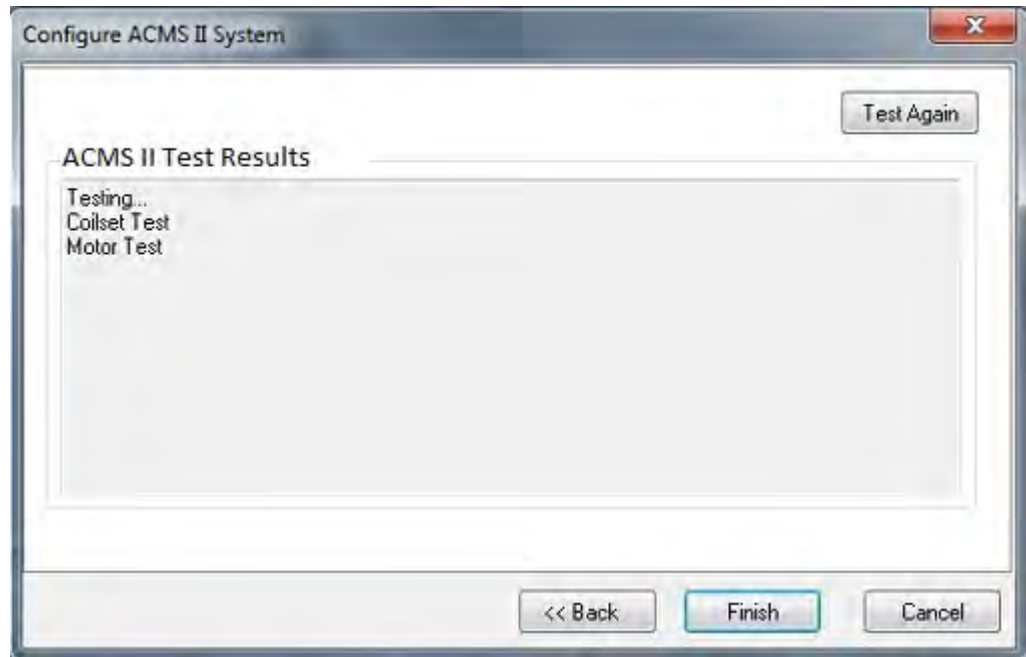


Figure 2-13. Configure ACMS II System dialog, page 2: Testing calibration file.

6. When the report in the **ACMS II System Test Results** area is complete and no errors are reported, click on the **Finish** button. The **Configure ACMS II System** dialog will close and you will be back at the **ACMS II Control Center** and the **Install** tab.
6. If you have completely followed the ACMS II hardware and software setup steps, the system is now ready for you to mount a sample, install it in the cryostat, and perform AC susceptibility and DC measurements, as is explained in Chapters 3 and 4. First, please review Section P.5, "Safety Guidelines and Regulatory Information" for important information.

2.3 Removing the ACMS II Option



WARNING!

Verify that there are no nearby sources of magnetic field (e.g., NMR or other laboratory magnets) before attempting to install or remove the linear motor transport, as explained in Section P.5.3.

You do not need to remove the sample linear motor transport and its associated hardware from the system while it is idle. However, if you intend to use it for other types of measurements (e.g., Heat Capacity, Thermal Transport), you must first remove the ACMS II option.

As summarized below, you will use the **ACMS II Install/Remove Sample Wizard** to prepare the system so that you can remove the sample linear motor transport, sample tube, and coilset puck. These procedures are essentially the reverse of the installation procedure.

Summary of ACMS II Removal Procedures

1. Prepare for removal of the sample linear motor transport and hardware:
 - a. Set the field to zero.
 - b. Use the **ACMS II Install/Remove Sample Wizard** to warm the sample chamber to 300 K, vent the chamber, and move the transport to the load position.
 - c. Remove the sample rod.
 - d. Shut down the sample linear motor transport.
2. Deactivate the ACMS II software application.
3. Remove the sample linear motor transport and place it in the storage case.
4. Remove the sample tube and the coilset puck.

2.3.1 Prepare for Removal

1. If necessary, activate the ACMS II software from MultiVu (select **Utilities >> Activate Option >> ACMS II**) as explained in Section 2.2.7.
 - If the field is not zero, set it to
 - Use the **ACMS II Install/Remove Sample Wizard** to remove the sample rod.

CAUTION!

Verify that you have removed the sample rod before continuing.


2. To continue with the ACMS II removal process, end the **ACMS II Install/Remove Sample Wizard** by clicking on the **Cancel** button. This button will close the install dialog and return you to the **ACMS II Control Center** and the **Install** tab. Before you can remove the sample linear motor transport, you must deactivate the ACMS II option, as is explained in Section 2.3.2.

2.3.2 Deactivate the ACMS II Option


1. Select **Utilities >> Activate Option** from the dropdown menu of the MultiVu window (Figure 2-9).
2. When the **Option Manager** dialog opens, select **ACMS II** and click on the **Deactivate** button. This will move the ACMS II option from the **Active Options** section of the dialog to the **Available Options** section. The **ACMS II Control Center** and the **ACMS II Log** window will close, but the MultiVu software application will remain open.
3. Continue with the ACMS II removal procedures below.

2.3.3 Remove the Sample Linear Motor Transport

1. Unplug the electrical connector from the back of the sample linear motor transport. (You can leave the other end of the cable connected to the Motor Module.)
Important: Never attempt to move the sample linear motor transport when it has a cable connected to it.
2. Remove the flange clamp from the top flange of the sample chamber (see Figure 2-7).

	WARNING!
<p>Be careful when lifting the linear motor transport. Do not drop the motor, because doing so could cause damage. Do not attempt to lift the motor by yourself, because doing so could cause injury. Take care not to damage the bottom and top "o" ring sealing surfaces.</p>	

3. Slowly lift the sample linear motor transport until it has cleared the stabilizer post (see Figure 2-7).
4. Place the sample linear motor transport back in the storage case (4096-150).

	WARNING!
<p>Store the sample linear motor transport in a secure location to prevent it from being attracted to magnetic fields in the laboratory, including those produced by the superconducting magnet, as explained in Section P.5.3.</p>	

2.3.4 Remove the Sample Rod Guide Tube and ACMS II Coilset Puck

1. Remove the sample rod guide tube from the sample chamber.

2. Remove the ACMS II coilset puck from the sample chamber by using the puck-extraction tool.²
3. Unplug the ACMS II drive cable from the probe head and set it aside. You do not need to disconnect the other end of the cable from the ACMS II Module.
4. Return the blank flange to the top of the probe head or install another of the Quantum Design measurement options.
5. When the sample chamber has been closed, you can purge and seal it by using the **Chamber** dialog box.
 - Select **Instrument >> Chamber**.
 - In the **Chamber** dialog box, click on the **Purge/Seal** button.
6. The base measurement system is now ready for you to install a different option.

² See Footnote 1.

Sample Preparation and Mounting

3.1 Introduction

This chapter contains the following information:

- Section 3.2 discusses constraints on the samples that can be measured with the ACMS II option.
- Section 3.3 explains how to mount samples for measurement with the ACMS II option.

3.2 Sample Properties

The quality of your AC and DC measurement results will be affected by the dimensions and shape of the sample and the size of its magnetic moment.

3.2.1 Size and Shape

The geometry of the detection coils in the ACMS II constrains the dimensions of samples that can be measured. Figure 5-4 gives the dimensions of the ACMS-II coilset puck. In order for the sample and sample holder to fit into the detection coils without a high risk of rubbing against the coil set bore, their diameter should be less than 6 mm. Frictional heating (especially at low temperatures) and noise in the measurements at high fields are common symptoms of friction between the sample holder and coil set. In AC measurements, the sample is vertically centered within one of the detection coils, so an accurate reading of the AC magnetic properties requires samples that are less than ~6mm high, i.e., that have a small vertical size compared to the 14 mm height of one of the detection coils. For DC measurements, in which the sample is vibrated halfway between the lower and upper detection coils, the reported DC magnetic moment is more sensitive to the sample size and shape than AC measurements. The DC response is calibrated against the included Pd standard, so accuracy of the reported DC moment will be ideal for a sample of these same dimensions. Since this measurement is identical to that used by the VSM option and these coils are larger than the VSM detection coils, the reported moment for DC measurements in ACMS-II will be less dependent on sample size and shape than in the VSM option (see Figure 3-1 in *VSM Option User's Manual 1096-100*).

3.3 Mounting Samples

3.3.1 Accurate Sample Location

The ACMS-II system uses a touchdown technique for automatic centering of the sample in the detection coils. This technique is described in detail in Chapter 4. To optimize the touchdown, take special care with two steps in your preparations:

- Verify that the sample is mounted on the sample holder near 25 mm from the bottom of the sample holder. Quantum Design has provided you with a special sample mounting fixture (see Figures 4-1 and 5-8) for locating the sample.
- Verify that the end of the sample holder has a very well defined contact surface for performing the touchdown operation.
- The total length of the sample holders should be 96mm: If using a straw, cut the straw to 83mm to give correct total length when assembled with the straw adapter (4084-815)

Quantum Design recommends that you locate the sample at an offset of 25 mm. The height above the puck surface of the bottom detection of the coilset is about 30 mm, so the offset of 25 mm leaves 5 mm of a safety gap. Note that for DC measurements there will be an “end effect” signal from the bottom of the sample holder to account for. This will appear as a diamagnetic (negative slope in $M(H)$) contribution for sample holder materials that are paramagnetic (e.g., straws).

3.3.2 ACMS Sample Mounting Techniques

1. The **sample holders supplied by Quantum Design** are designed to fit on the mounting station. The total length of the holder and exact location of the sample are better defined by using the mounting station. While the fragile quartz paddle is designed for the lowest moment samples, a more robust straw provides versatility.
 - A. The **quartz paddle** is redrawn from a 4 mm diameter rod. Elimination of surface microcracks improves mechanical strength of the quartz paddle. A nitric acid dip removes surface impurities. Smooth surface can make cleaning easy but reduces adhesion of glue. The adapter is made of glass-filled polycarbonate, which should never be exposed to organic solvents, especially acetone. The outer gluing surfaces of the quartz pieces are sanded to help bind with the epoxy. A complimentary shaped shim is added to the paddle and put inside the adapter containing epoxy resin. The epoxy is specially chosen to withstand both high temperatures (400 K) and cryogenic conditions. The assembly is cured straight in a custom fixture.



- a) For mounting **thin films parallel to field** (see figure 3-1 to the left), do not exceed the width of the paddle. Wider samples can be mechanically secured and better protected using the brass trough or aluminum frame techniques. Place the quartz holder in mounting station. Using a sharp wooden stick or other tool, place a small drop of glue on the paddle, set film on top and press to secure bond. Let it dry completely at ambient conditions. If the sample can handle the heat, increase temperature to 340 K and purge sample chamber. Return to 300 K, purge and seal, or lower temperature and follow centering routine. To remove sample, hold length of quartz paddle with no pressure on adapter junction. A thin wooden flat can transport solvent under the chip. Wait for solvent to penetrate. Free the sample with appropriate leverage.
- b) For **single crystal** samples and low temperature analysis, put GE 7031 on crystal then secure to quartz. A small amount of **fine powder** can be mixed into a drop of varnish, although it is difficult to get a quantitative mass with this technique. If taken to 340 K to cure before completely dry, a large field like 5 Tesla should result in the alignment along a preferred crystallographic axis which is frozen in place by going cold. Kapton tape is also effective way to secure small amounts of material, but keep sample as point source and tape symmetrical along the axis of motion. Concern with tape is the random contamination from dust in a lab environment.
- c) To **clean the quartz paddle** use a solvent specific to the glue. Cotton swabs help keep all solvents away from the polycarbonate adapter, especially acetone. Do not use sonicator, which could introduce cracks at the junction points. The common break point is at the adapter junction and caused by lateral force on the sample rod. Frequently, breakage occurs during the sample mounting and cleaning process.

Figure 3-1. Quartz Paddle.

B. Clear plastic Straw and adapter

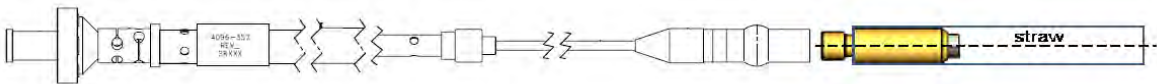


Figure 3-2. The ACMS II Sample Rod (4096-352) and Straw Adapter (4084-815).

Quantum Design provides a specific type of clear drinking straws (QD part # AGC2-BOX) that are held securely using a straw adapter (p/n 4084-815). The 2 part adapter works by pushing the straw over the barb of the main body (much like in older straw barb adapters from QD) and then screwing the collar down to retain the straw. Use straws for the following samples:

- a) Films perpendicular to field: diagonal size = 5.8mm (e.g., 4.1 x 4.1mm square film); this is best done by placing the film near the recommended 25mm offset and using two clean applicators from each end of straw to turn it sideways and jam it into the straw material so that it does not move.
- b) Films parallel to the field: width = 6mm; this width will ensure that the film does not slip, but note that if precise vertical alignment of the film is required, the quartz paddle may be preferred

For bulk samples or films that are slightly too small, consider the technique of slitting a ~80mm section of straw lengthwise so that it can be inserted in another straw, providing a smaller inner diameter for sample mounting. To immobilize the inner straw after it is inserted in the main

straw, poke several holes with a pin near the bottom of the assembly so that the holes pass through both the outer and inner straws.

The **palladium standard** is mounted with GE-7031 varnish to help withstand thermal cycles during installation tests. It is mounted in the brass sample holder used for VSM option. The brass holder is not used for ACMS-II due to the AC susceptibility of the brass, but since the Pd is strictly a DC standard this is not an issue. The sample offset of the Pd sample is also 35mm in order to minimize the sample holder end effect. The only function is DC calibration of magnetometer at 1 Tesla and 298 K. To get the expected moment, simply multiply the mass of palladium, the applied field and the susceptibility, which is 5.25×10^{-2} emu/gram-Tesla at 298 K. The expected moment at 1 Tesla for a 0.25 gram cylindrical shape is 0.013 emu. Since the magnetization versus temperature for palladium has characteristic features, it is not a suitable thermal reference standard. Consider temperature independent diamagnetic Quantalloy or the Curie-Weiss paramagnet dysprosium oxide pellet. Even at the lowest temperatures, the contribution from impurities in the varnish is below 0.1% of the large Pd moment.

C. Sample cleaning supplies

Specific cotton swab for brass trough iso-propanol (IPA), acetone, toluene, nitro-methane



Figure 3-3. Sample cleaning supplies.

D. Just a few of the **methods and materials for securing the sample** to the holder are presented. The primary goal is to keep the sample a point source dipole during the measurement. Since any glue or tape will introduce point source dipole contributions to the total magnetic moment, the material should be applied sparingly and symmetrically.

1. The term **glue** is loosely applied to any material that will secure a sample to a support material. Generally the materials will harden over time, with the process aided by increasing temperature and keeping under vacuum. Pockets of trapped oxygen or adsorption to very high surface area materials will produce a magnetic signature around 50 K. Each material has a specific range of conditions for optimal usage; with varnish best at the lowest temperatures and Duco cement the most easily removed. Since the contents of each glue are not normally disclosed by manufacturers, it is hard to know magnetic properties. A general rule is that colorless materials are less magnetic than colored

ones because dyes used in the materials tend to have magnetic signature. However, it is best for users to verify the magnetic properties of the glue.

- a) The legendary **GE7031 varnish** is a vinyl phenolic adhesive (safety labeling H=1; F=4; R=3; PE=3). As the bulk material in container ages, it will become thicker. Adding iso-propanol (IPA) and/or toluene will thin solution. Cleaning is best aided with toluene. The aging and/or oxidation process will lead to a darker coloring of the solution. This tends to correlate with increased magnetic signature. The ideal solution is a light tan honey colored solution, freely flowing without addition of extra solvents.
 - b) Easily obtained **Duco cement** is useful at room temperature and for securing materials of similar thermal properties, like quartz on quartz. Differing materials will force cracking in the brittle Duco cement upon cooling below 150 K. Duco may go on thick but it will dry or cure to a thin residue. A key reason for regular use is Duco solubility in acetone. Powders secured with Duco can be easily recovered by soaking in acetone.
 - c) Easily obtained **Superglue** (cyanoacrylate) is a fast drying, very secure bonding method for room temperature measurements. The very thin nature allows low mass, low magnetic signature application. At the colder temperatures, it may not hold materials with different thermal properties. The solvent of choice is nitro-methane. Still readily available as a stock car fuel, it should be handled carefully. A cotton swab of nitro-methane with a thin stick to wedge the sample off the holder should work effectively. Please see MSDS for proper ventilation requirements when using these solvents.
2. Tape is a simple and effective method for securing to the quartz paddle. It will prevent the sample from being lost during the measurement. The background can be reasonably small if the mass is symmetrical about the sample. Keeping the tape clean from magnetic dust around the lab is the primary concern.
- a) **Kapton tape** is commonly used at low temperatures and is available from Quantum Design. It retains properties over entire range of 1.8 K to 400 K. As a point source may show 10^{-6} emu ferro-magnetic signature at room temperature. Like with a straw, if the tape is evenly distributed in the background, there will be no contribution to the reported moment of the sample. To replace common drinking straws, Kapton tubing is available through the medical community and may provide flexibility in diameter selection with lowest level of impurities. Organic solvents like toluene or maybe iso-propanol (IPA) can help dissolve the glue remaining on the holder after tape is removed.
 - b) Thin Teflon tape can be wrapped around the quartz holder to secure the sample location without use of glue. However, the technique tends to produce a significant paramagnetic oxygen peak around 50 K. As a precaution, add a heat and purge step to the sequence. The Teflon tape can cover the full temperature range of the instrument, but 350 K is usually sufficient for this purpose. When Teflon becomes a point source dipole like when wrapping the sample, a more significant ferro-magnetic contribution is measured. Each batch should be tested for impurities.
 - c) The standard Parafilm roll and various wax materials can be used to secure the sample. Watch maximum exposure temperatures of the wax. These types of materials are typically low moment background and useful with air-sensitive samples or when recoverability is desired.

Immediate Mode Measurements

4.1 Introduction

This chapter contains the following information:

- Section 4.2 describes the AC and DC measurement processes and compares these two measurement types.
- Section 4.3 explains how to install and center the sample.
- Section 4.4 explains how to perform AC and DC measurements.

4.2 Overview of ACMS II Measurements

The ACMS II is both an AC susceptometer and a DC magnetometer. The distinction between the two types of measurements is important. DC magnetization measurements measure a sample's magnetic moment in an applied magnetic field at a specific temperature. AC susceptibility measurements do not directly measure a sample's magnetic moment but are far more sensitive.

Once the ACMS II is electronically connected to the system, it is fully configured to perform both AC susceptibility and DC magnetization measurements. No hardware or software reconfiguration is necessary to switch from AC susceptibility to DC magnetization. In fact, you can actually perform both measurements on a sample within a single sequence. To save time, both AC susceptibility and DC magnetization can be measured at each measurement step. If the magnetization criteria are different from the susceptibility criteria, the sequence can be configured to perform each measurement individually, yet within a single sequence.



Before a sample is measured for the first time, it should be centered relative to the detection coils that are in the ACMS II insert. Section 4.3 discusses centering methods.

4.2.1 Choosing a Measurement Method

In the ACMS II option, AC measurements are nearly three orders of magnitude more sensitive than DC measurements. Conductive samples usually yield much larger signals during AC measurements. However, in some situations, DC measurements produce better results than AC measurements. For example, weakly paramagnetic samples with magnetic moments near the noise floor of the ACMS II's AC measurement capability ($\sim 1 \times 10^{-8}$ emu) can often be detected more easily in the large DC fields applied by the PPMS superconducting magnet, because the larger DC field raises the sample's moment well above the noise floor for DC measurements ($\sim .5 \times 10^{-6}$ emu). In such cases a DC center (section 4.3.3) is appropriate. Most PPMS platforms contain at least 7-T DC magnets, while the ACMS II can only apply alternating fields of up to ± 10 Oe consistently (1 T = 10,000 Oe). In addition, large or asymmetric sample holders generally require DC methods to properly account for background sources. Other symmetry and field uniformity concerns—such as small applied fields—usually demand DC methods as well.

There are many other points to consider when determining whether to use AC or DC centering and measurement methods. It is your responsibility to choose centering and measurement techniques that are appropriate for the type of information being sought and the type of sample being examined. Important sample characteristics to consider include type of magnetic behavior, magnetic moment, conductivity, shape, and size. Applied field and temperature can also be important. Finally, the nature of the information that the experiment examines should be considered. AC susceptibility measurements are fundamentally different than DC measurements in that they report a *change* in a sample's magnetic moment with a relatively small change in field, rather than a sample's absolute moment in a relatively large constant field. Contact a Quantum Design representative if you would like to discuss considerations specific to your application.

4.2.2 Operating Modes

ACMS II measurements and sample centering may run in immediate mode or sequence mode. ACMS II control center commands and PPMS MultiVu **Measure** menu commands execute operations immediately, but each control center or menu command must be selected manually. ACMS II measurement sequence commands included in a PPMS MultiVu sequence file are executed automatically when they are read while the sequence runs. Any number and combination of measurement sequence commands may be included in a sequence file.

Data from immediate-mode measurements or sample centering is saved only if an ACMS II measurement data file is selected to store the data. Measurement and selected system data read during sequence mode measurements and sample centering is automatically saved to the specified data file.

The immediate-mode measurement dialog boxes show the results from the last measurement of that same type (see figure 4-5, for example). The sequence mode measurement dialog boxes do not show measurement results, but you can use them to select which system data items you want to save to the specified ACMS II measurement data file.



You are encouraged to use the ACMS II control center to perform all normal system operations. The automated routines in the control center help ensure that you complete the necessary procedures when you install new samples and create data files. This chapter illustrates use of the control center.

The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses sequence files, sequence operation, and all standard PPMS sequence commands in detail.

4.3 Installing a Sample

4.3.1 Attach a Sample and Measure the Sample Offset

1. Attach the sample to the sample holder using the techniques discussed in Chapter 3.
2. Use the sample-mounting station to measure the distance from the center of the sample to the bottom of the sample holder, reading the position from the scale as demonstrated in Figure 4-1. This distance is called the sample offset. Measure the sample position to an accuracy of 0.5 mm. The ACMS II hardware can safely accommodate a sample offset between zero (i.e., sample is at the bottom end of the sample holder) and 25mm. However, a sample offset less than ~20mm can result in a significant signal from the magnetic “end effect” of the sample holder. A sample offset larger than 25mm presents the risk of the sample holder rattling against the puck surface when measuring.

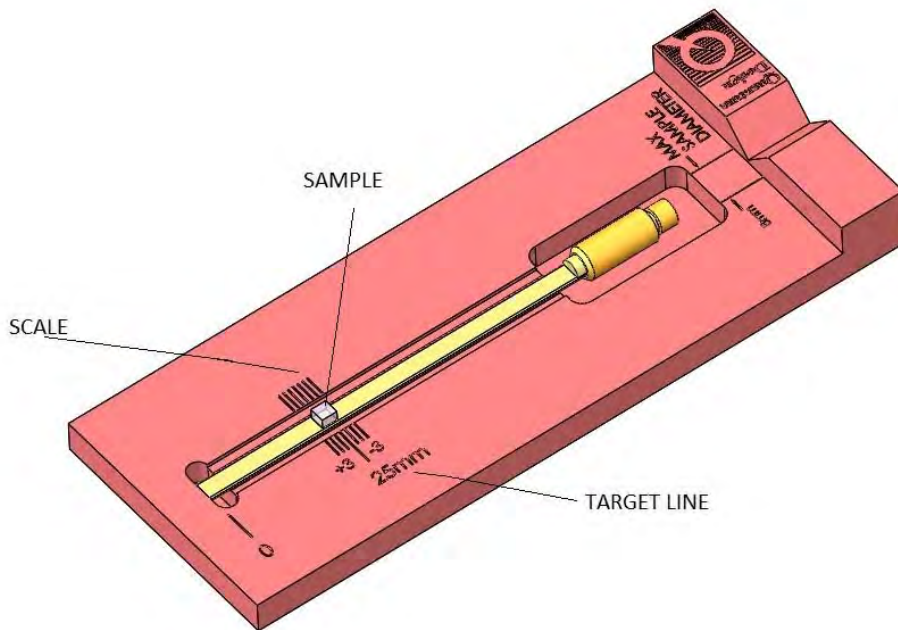


Figure 4-1. Reading the position of the sample from the sample-mounting station

3. Remove the sample holder (with the mounted sample) from the mounting station and screw it firmly onto the end of the sample rod.
4. Inspect the sample rod and sample holder to ensure they are straight. Deviations can result in rubbing of the sample or sample holder in the coil set which causes heating at low temperatures and noise in the measured moment when a magnetic field is applied.

4.3.2 Activate the ACMS II Option and Control Center

1. To start the ACMS II application and open the **ACMS II Control Center**, select **Utilities >> Activate Option** in the MultiVu window.
2. At this time, the system also will perform a **Home** operation to determine the full range of travel for the sample transport by touching down and then going to the load (top) position.

4.3.3 Install the Sample

1. In the **Install** tab of the **ACMS II Control Center**, click on the **Install/Remove Sample** button.
2. Click on the **Open Chamber** button (below the **Instructions** area). The wizard will bring the sample chamber to room temperature, vent the chamber, and move the transport to the load position, and the **Instructions** area will show the status of these processes.
3. When the **Instructions** area indicates that the chamber has been flooded and the transport is in the load position use the sequence outlined below to install the sample rod and sample.
 - If you have not already measured your sample offset position, use the sample-mounting station to obtain the sample offset from the end of the sample holder (see Section 4.3.1).
 - Attach the sample holder to the sample rod.
 - Remove the cap from the top of the sample linear motor transport.
 - Insert the sample rod with the attached sample holder into the sample access port until the magnetic lock at the top of the sample rod engages the magnetic lock ring in the linear motor transport. Verify that the magnetic lock has engaged the magnetic lock ring.
Important: The sample will be subject to vertical magnetic fields of up to approximately 200 gauss when it passes through the head. If this is unacceptable for your samples, please contact your local Quantum Design service representative.
4. Click on the **Next >>** button at the bottom of the **ACMS II Install** wizard.
5. Open a new or existing output data file. Note that all parameters entered here are for informational purposes only and are not used in calculating the reported sample moment.
6. Click on the **Next >>** button at the bottom of the dialog. Here you can **Scan For Sample Offset** or **Enter Offset Manually** if you used the sample mounting station to visually locate the sample.
 - Select between AC and DC centering scan from the popup menu.
 - A sample signal larger than $\sim 10^{-5}$ emu is required in order to be detected using a DC centering scan. This may require the application of magnetic field in order to magnetize your sample.
 - Magnetic contamination and sample holder end effects can obscure the sample signal, so it is recommended to verify the scan results with the known approximate sample offset of the sample.
 - The **Advanced Centering** mode will locate the motor at a fixed height and will not permit touchdown centering. Thus, it is not generally recommended.

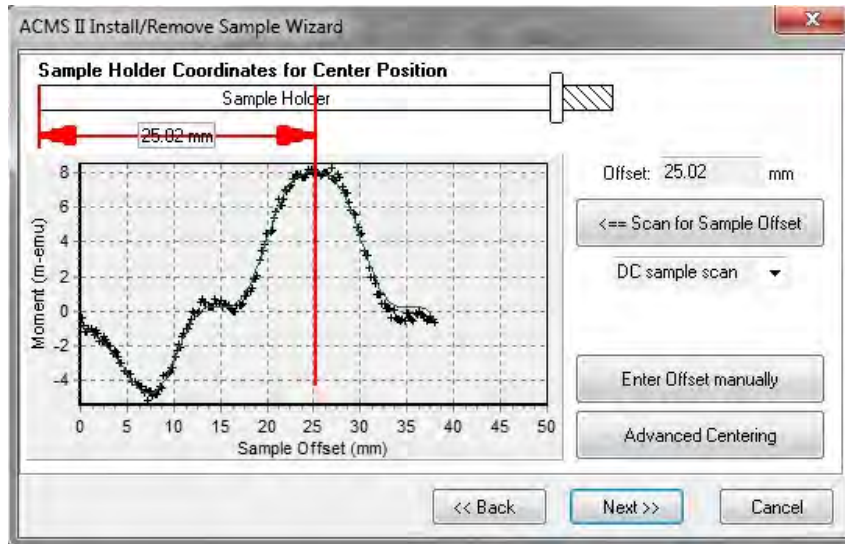


Figure 4-2. Example of a DC centering scan for a sample with a large positive magnetic moment.

7. Click on the **Next >>** button at the bottom of the dialog (Figure 4-2). This button opens the last page of the **ACMS II Install** wizard (Figure 4-3), which reports the sample-offset position and related instructions.

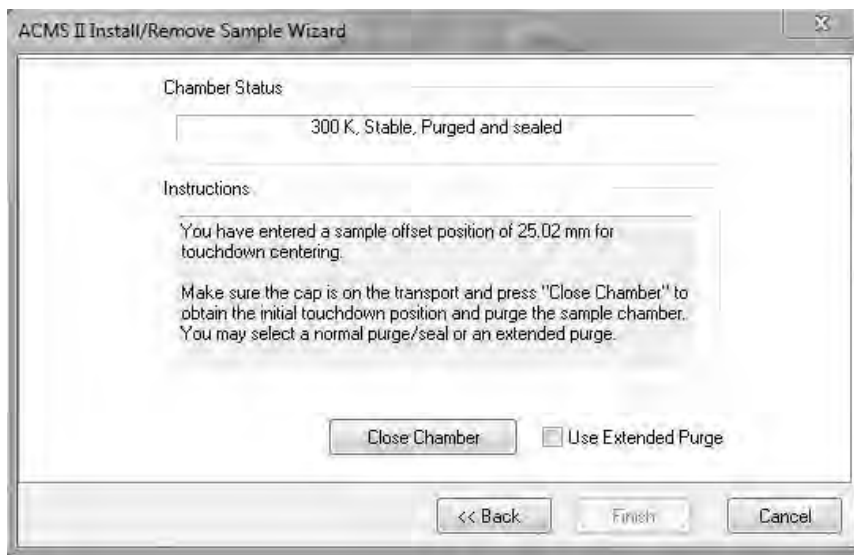


Figure 4-3. The ACMS II Install wizard, final page

Select **Use Extended Purge** any time you plan to use low temperatures ($T < 270$ K). Note that this ~10 minute extended purge must complete before measurements of temperature variations are started.

1. Place the cap on the linear motor transport and click on the **Close Chamber** button.
IMPORTANT: do not perform any CPU-intensive tasks on the computer while the touchdown operation completes.
2. You can now set up the system to perform sample measurements.

4.3.3.1 THE TOUCHDOWN OPERATION

Automatic sample centering is accomplished with the touchdown operation. To perform a touchdown operation, the linear motor transport lowers the sample rod until the end of the sample holder touches the puck surface (see Figure 4-4). At this point, the software knows the precise offset between the coilset and the sample, based on the dimensions of the coilset and the location of the sample on the sample holder. The linear motor transport then moves the center of the sample to the center of the coilset to continue measuring according to the following relation:

$$\text{Measure location} = (\text{touchdown location}) + (\text{coilset height}) - (\text{sample offset}),$$

where coilset height ("H" in Figure 5-4) is a calibrated constant for a given puck and is near 40mm.

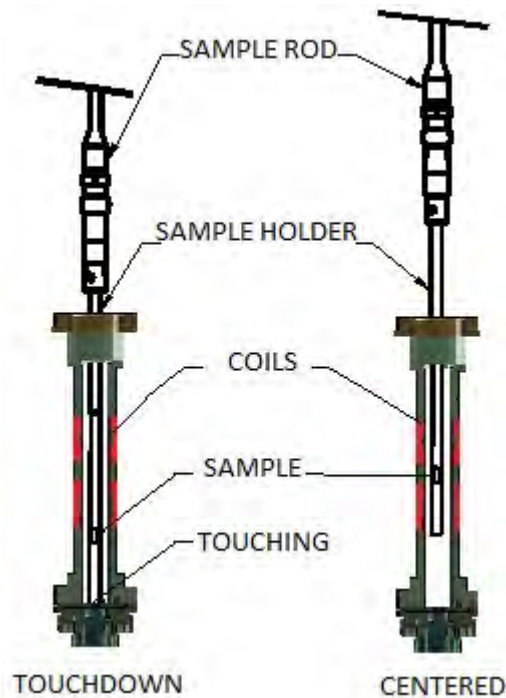


Figure 4-4. The touchdown centering operation

4.3.3.2 SCHEDULING TOUCHDOWN OPERATIONS

The software usually performs touchdown operations automatically at prescribed intervals of temperature (typically 10 K) or time (typically 10 minutes). Touchdowns can also be performed explicitly in a sequence (**DC >> Center Sample**) in immediate measurement mode (**Measure >> DC Center Sample** in the MultiVu dropdown menu)

CAUTION!

Failure to perform frequent enough centering operations will lower the accuracy of measurements.

Automatic centering is a key quality-control mechanism in the software. When automatic centering has been disabled, the software cannot know how far the sample has drifted from the center of the pickup coils unless a touchdown is performed.

4.4 Taking ACMS II Measurements



ACMS II measurements can be taken only if the ACMS II hardware is installed and the ACMS II option is activated in the PPMS MultiVu software. Refer to chapter 2 to install and activate the ACMS II option.



A sample should be centered relative to the detection coils in the ACMS II insert before it is measured for the first time. A sample should be re-centered following any large temperature change, and it is advisable to re-center the sample prior to re-measuring it with a different measurement method. Refer to section 4.3.2 to center the sample.

Before you take measurements, you may want to refer to section 4.2.1, “Choosing a Measurement Method,” and section 4.2.2, “Operating Modes.”

4.4.1 Taking AC Susceptibility Measurements

4.4.1.1 TAKING A MEASUREMENT IN IMMEDIATE MODE

To summarize, you take a measurement in immediate mode by first installing the sample and then defining and running the measurement. Complete the following steps:

1. Select the **Datafile** tab in the ACMS II control center if you want to select a different data file or create a new file (see section 6.4.1.2). The **Properties** tab in the control center displays the sample property information stored in the header of the selected data file. If you run the measurement when no data file is selected, the data is discarded.
2. Mount the sample on a suitable sample holder. Refer to section 3.2.
3. Select the **Sample** tab in the ACMS II control center. Then select the **Change** button and insert the sample into the sample chamber. Section 3.3 describes this procedure.

4. Select the **Measure AC** button in the ACMS II control center *or* select the **Measure>ACMS II AC Susceptibility** menu option. The **ACMS II AC Susceptibility** dialog box opens (see Figure 4-5).

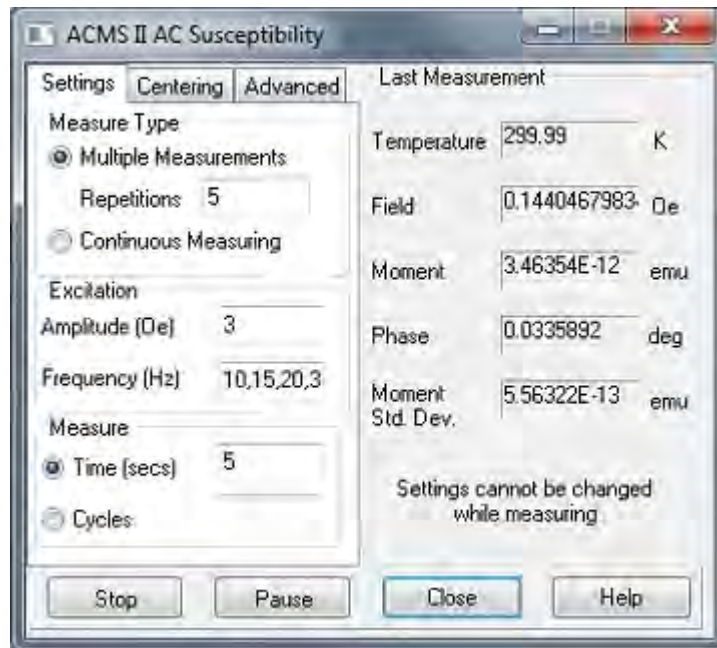


Figure 4-5. **AC Susceptibility** dialog box. Items on the left side of the dialog box designate measurement conditions that should be set prior to initiating the measurement. Items on the right side of the dialog box display the results of the last immediate-mode AC measurement.

5. Define the AC measurement as follows.
 - a. In the **Advanced** Tab, select the **AC Measurement Type** that defines the number and location of the individual measurements that are taken and averaged to obtain a single, final measurement value. Three measure modes are available for immediate-mode AC measurements. The default and most comprehensive measure mode is the five-point measurement. The fastest and least comprehensive mode is the one-point measurement. Refer to table 4-1.
 - b. In the **Settings** Tab, define the **Amplitude** of the sine wave excitation that is applied to the sample. Not all amplitude values can be achieved in all frequency and temperature ranges. Amplitudes up to 10 Oe are producible at all frequencies and temperatures.
 - c. In the **Settings** Tab, define the **Frequency** of the sample excitation signal.
 - d. Also in the **Settings** Tab, define either the measurement's **Time (secs)** or **Cycles**. **Time (secs)** specifies the boxcar average duration for each measurement and will be an integer number of period of sine wave excitation. **Cycles** on the other hand, specifies the number of excitation periods that elapses between recorded measurements.

Table 4-1. Measure Modes for AC Susceptibility Measurements

MEASUREMENT MODE	WHERE MEASUREMENTS ARE TAKEN	SUGGESTED USE
Five Point	(1) Center of coil array (2) Bottom detection coil (3) Top detection coil (4) Bottom detection coil (5) Center of coil array	This provides a calibrated measurement with temperature drift compensation. The center measure before and after the bottom-top-bottom measures cancels the effect of a linear temperature drift.
Three Point	(1) Bottom detection coil (2) Top detection coil (3) Bottom detection coil	This provides faster calibrated measurements without temperature correction. This is the default and should be good for most measurements.
One Point	(1) Bottom detection coil	Use for fastest, but least accurate, measurements. One-point measurement uses calibration from previous four- or five-point measurement.

e. Select the **Measure** button in the **AC Magnetization** dialog box. The measurement begins.

4.4.1.2 LIMITATION OF THE APPLIED AC FIELD DUE TO EDDY CURRENT HEATING

At low temperatures, the AC field created by the ACMS II can cause the PPMS sample and the ACMS II insert to warm. The AC field induces eddy currents in the copper portion of the sample chamber, which results in joule heating. In order to prevent such warming, the user should limit the applied AC field at temperatures below 25 K. Table 4-2 shows the maximum applied fields as a function of temperature, frequency, and measurement time that should be used to ensure a temperature rise of less than approximately 0.25%. Larger fields may be applied if more temperature rise is tolerable. Note that 17 Oe is approximately the maximum field amplitude the ACMS II can generate.

Table 4-2. Maximum AC Field Amplitude (Oe) to Avoid Warming

Temperature (K)	Frequency (Hz)				Excitation Field (Oe)
	10	100	1000	10000	
10	17	17	17	11	
4	17	17	17	7.7	
2	11.9	9.4	6.8	1.7	
1.9	9.4	6.8	5.1	0.85	
1.8	5.1	5.1	4.3	0.85	

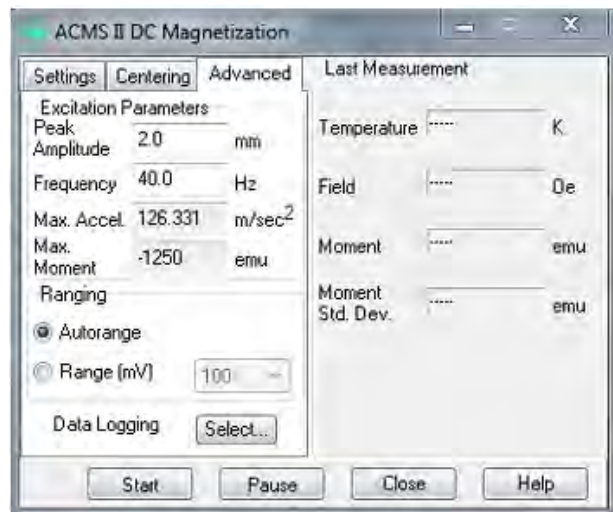
4.4.2 Taking DC Magnetization Measurements

4.4.2.1 TAKING A MEASUREMENT IN IMMEDIATE MODE

To summarize, you take a measurement in immediate mode by first installing the sample and then defining and running the measurement. Complete the following steps:

1. Select the **Datafile** tab in the ACMS II control center if you want to select a different data file or create a new file (see section 6.4.1). The **Properties** tab in the control center displays the sample property information stored in the header of the selected data file. If you run the measurement when no data file is selected, the data is discarded.
2. Mount the sample on a suitable sample holder. Refer to section 3.2.
3. Select the **Sample** tab in the ACMS II control center. Then select the **Change** button and insert the sample into the sample chamber. Section 3.3 describes this procedure.
4. Select the **DC Measure** button in the ACMS II control center *or* select the **Measure** → **ACMS DC Magnetization** menu option. The **DC Magnetization** dialog box opens.

Figure 4-6. **DC Magnetization** dialog box. Items on the left side of the dialog box designate measurement conditions that should be set prior to initiating the measurement. Items on the right side of the dialog box display the results of the last immediate-mode DC measurement.



5. Define the DC measurement as follows.
 - a. Define the peak amplitude and averaging time to obtain a measurement value.
 - b. Select the **Ranging** mode used for voltage detection. **Autorange** changes the gain setting at the beginning of every measurement so that the optimum setting is selected. **Sticky Autorange** prevents the gain from changing until it is absolutely necessary. **Fixed Range** uses only a single user-specified gain setting.
6. Select the **Start** button in the **DC Magnetization** dialog box. The measurement begins.

ACMS II Hardware

5.1 Introduction

This chapter contains the following information:

- Section 5.2 describes each of the basic hardware components that make up the ACMS II option.
- Section 5.3 describes the contents of the ACMS II User's Kit.
- Section 5.4 describes the sample-mounting station and its use.
- Section 5.5 describes the electrical components of the ACMS II option.

5.2 ACMS II Hardware Components

This section describes each of the basic hardware components that make up the ACMS II system. For instructions about installing the various components, please refer to Chapter 2.

5.2.1 Sample Transport

The sample linear transport (4096-400) is the motor that moves the sample. The linear motor transport and many of its component parts are illustrated in Figure 5-1. You will mount the linear motor transport directly on top of the PPMS, DynaCool, or VersaLab sample-chamber opening after you have inserted the sample tube into the sample chamber.

The sample rod is inserted into the sample chamber through an access port on top of the sample linear motor transport. The sample chamber is sealed (for storage and during operations) by a cap and an O-ring seal. An electrical connector at the rear of the linear motor transport provides the Model CM-A with electrical access to the drive coil and position encoder via the motor drive cable (3096-200 or 3096-201).

The sample rod is held in place in the sample linear motor transport by a magnetic-locking mechanism consisting of small magnets in the top of the rod; the magnets stick to a thin steel ring at the top of the armature.

A spring-suspension mechanism inside the linear motor transport isolates the vertical motion of the motor from the housing during vibration. The resonant frequency of the spring-suspension mechanism is about 5 Hz. You can verify whether the suspension mechanism is functioning

correctly by gently tapping the top of the sample rod at the sample access port during sample loading. A mechanism that is working correctly will oscillate for at least 5 seconds.

The transport is shipped from the factory with the shipping plug installed to prevent oscillation of the motor suspension mechanism. Before using the equipment, you must remove the shipping plug and install the extender tube flange.

A window in the front side of the linear motor transport (opposite the electrical connector) displays the location of the armature (the moving section of the motor transport). When you are installing or removing samples, you will be able to see an indicator pin at the top of the window: The indicator pin will be at the *top* of the window when the system is in the “load” position. The indicator pin will be at the *bottom* of the window, which is the “shutdown” position, *only* when the sample rod has been removed and the linear motor transport has been shut down. During AC measurements the indicator pin will move to pre-determined positions and stand still for a few seconds while for DC operations, the indicator pin will vibrate rapidly (1–4 mm peak-to-peak amplitude at 40 Hz) between the load and shutdown positions.

Important: The sample will be subject to vertical magnetic fields of up to approximately 200 gauss when it passes through the head. If this is unacceptable for your samples, please contact Quantum Design.

CAUTION!

- ◆ The indicator pin should be at the bottom of the window when the linear motor transport has been shut down. If it is NOT, it indicates that the sample rod has been inadvertently left in the sample chamber.
- ◆ Never attempt to remove the linear motor transport from the system while the sample rod is installed.

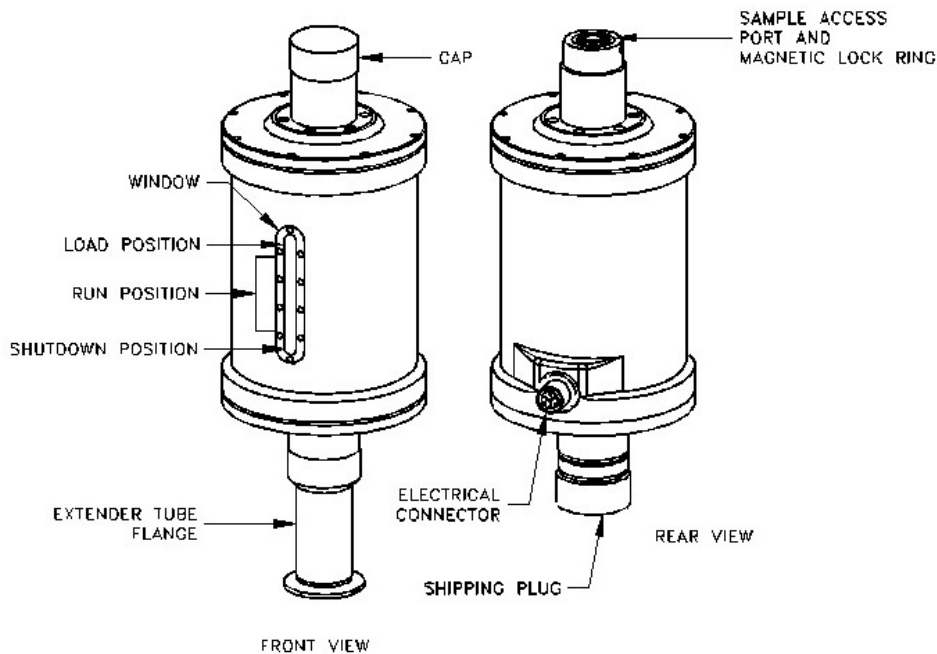


Figure 5-1. Sample linear motor transport (4096-400)

5.2.2 Storage Case for Linear Motor Transport

Because the linear motor transport is both heavy and delicate, Quantum Design has furnished you with a specially designed storage case to protect it during transport and storage. When the linear motor transport is not being used, it should be stored in this case. You can keep the case on a shelf or in a cabinet, so long as it is on a stable base and is stored upright as shown in Figure 5-2.

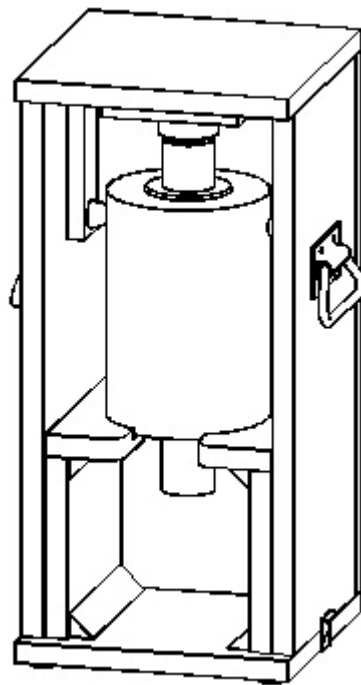
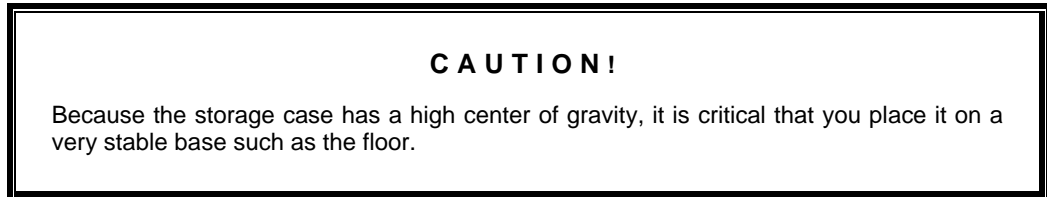


Figure 5-2. Storage case (4096-150) for the linear motor transport.

5.2.3 ACMS II Coilset Puck

The coilset puck contains the ACMS II detection coils and a thermometer for monitoring the sample temperature. You will insert the coilset puck into the sample chamber by using the PPMS sample insertion tool (also called the puck insertion/extraction tool) and the same procedures used to insert other types of pucks (see the *Physical Property Measurement System: Hardware Manual* for information on puck insertion and extraction). You must install the puck *before* you insert the sample tube.

The electrical connector at the bottom of the coilset puck has a serial number (see Figure 5-3). As explained in Sections 2.2.3, you will use this serial number to verify the numbers contained in the application software. The system uses the serial number to identify the puck calibration information.

The dimensions of the standard coilset puck are shown schematically in Figure 5-4. The center of the gradiometer pick-up coils is located 40.1 mm (1.58 in) above the location corresponding to the puck surface. This position corresponds to the center position of the magnet.

After repeated insertions of the ACMS II puck, the contact fingers at the base of the coilset (above the serial number in Figure 5-3) might bend inwards, which would loosen the fit of the coilset in the bottom of the sample chamber. When this happens, you should make the coilset fit snugly again by using the puck adjustment tool, as described in the *Physical Property Measurement System: Hardware Manual*.

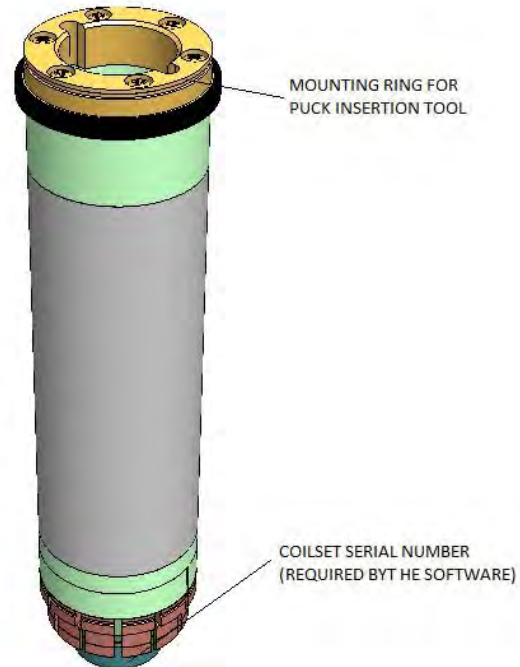


Figure 5-3. ACMS II Coilset puck (4084-850)

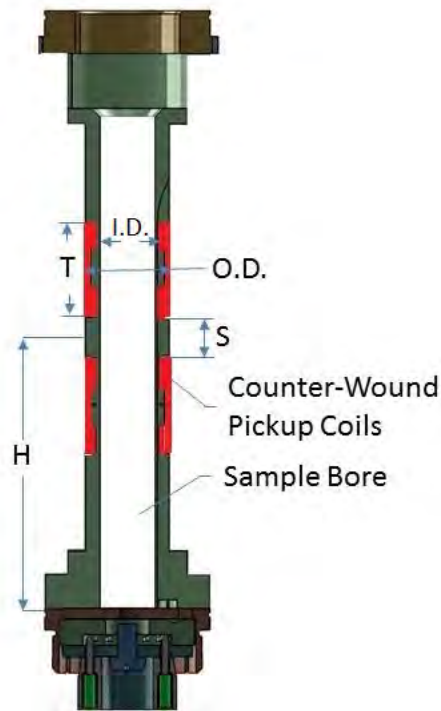
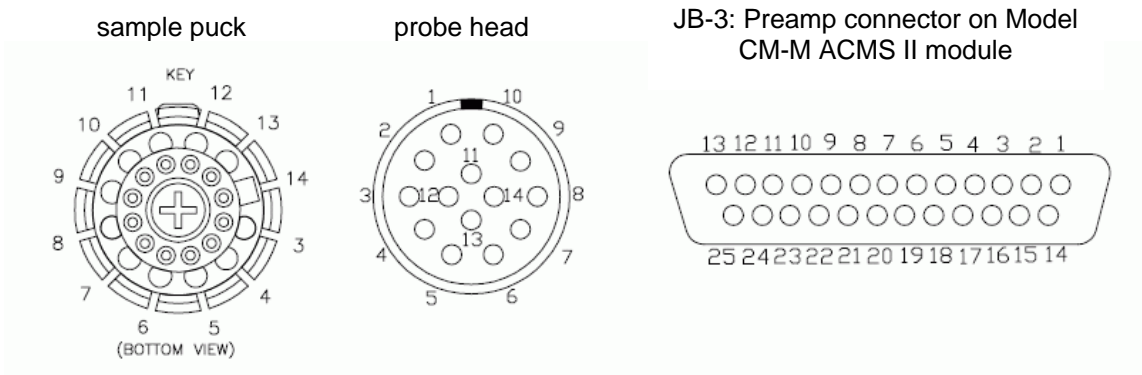


Figure 5-4. The internal dimensions of a standard coil set puck are as follows: Bore Inner Diameter (I.D.) = 8.1 mm; coil thickness (T) = 14.0 mm; coil spacing (S) = 5.9 mm; coil outer diameter (OD) = 12.4 mm; height above puck surface (H) = 40.1 mm.

Table 5-1. Sample Connection with ACMS II Detection Cable Connected.



SAMPLE PUCK	GRAY LEMO CONNECTOR AT PROBE HEAD	JK-1 SIGNAL ON CM-M ACMS II MODULE	FUNCTION
3	3	14	Thermometer Current +
4	4	7	Thermometer Current –
5	5	15	Thermometer Voltage +
6	6	8	Thermometer Voltage –
7	7	1	Trim Coil +
8	8	9	Trim Coil -
9	9	4	Calibration Coil -
10	10	12	Calibration Coil +
11	11	5	Detection Coil +
12	12	13	Detection Coil –
13	13	3	Drive Coil +
14	14	10	Drive Coil –

5.2.4 Sample Rod Guide Tube

You will insert the sample tube (Figure 5-5) into the sample chamber after you have installed the coilset puck. The sample tube provides low-friction guide sleeves for the sample rod. The top of the sample tube consists of an integrated O-ring attached to a stabilizer post. The stabilizer post provides a rigid, bayonet-style mount that prevents the linear motor transport from tipping over. Part of the post extends into the sample chamber and part extends into the extender tube flange on the bottom of the linear motor transport, as shown in Figure 2-7.

CAUTION!

You should always use the flange clamp to hold the linear motor transport onto the post, even though the stabilizer post helps prevent the linear motor transport from tipping over.

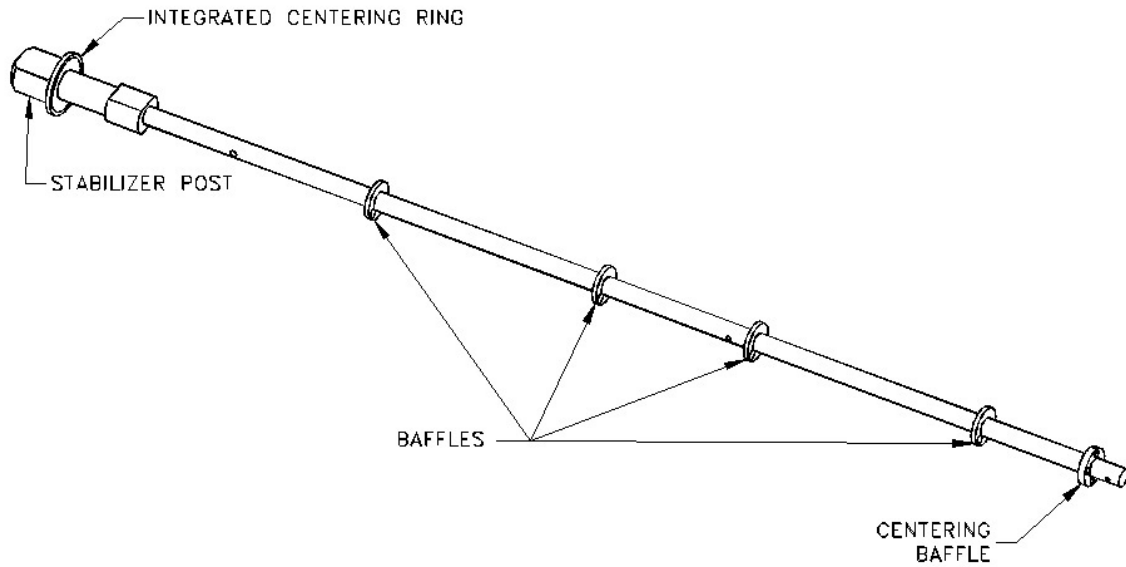


Figure 5-5. Sample rod guide tube (4096-301 shown for example)

5.2.5 Sample Rod

Many of the design features of the sample rod were introduced to reduce the background signal from mechanical vibrations at the DC oscillation frequency. Such features control the rattling or friction between the rod and the system and they eliminate resonant flexure of the shaft.

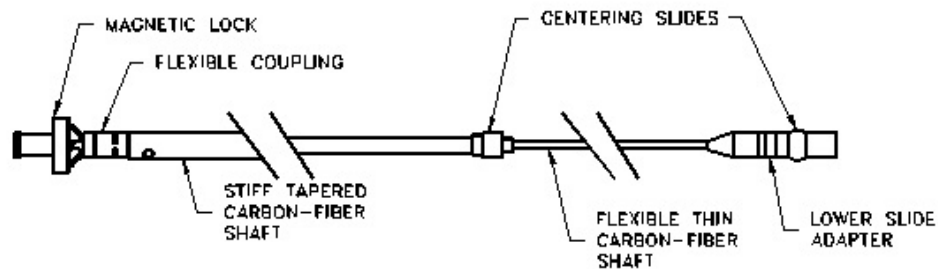


Figure 5-6. ACMS II sample rod (4096-352 shown for example)

Figure 5-6 shows the sample rod in a horizontal position (during use, the magnetic lock would be at the top of the rod).

Important: Keep the magnet surfaces clean, as the strength of the lock depends on the magnets being flush in contact with the mating part in the linear motor transport. Also, avoid bringing the magnets into contact with magnetic objects. Although small, the magnets are extremely strong.

5.3 ACMS II Option User's Kit

The ACMS II Option User's Kit contains miscellaneous hardware and supplies that you will use to mount samples and to verify the operation of the option. The convenient portable toolbox (see Figure 5-7) helps organize the items.

The kit includes the following contents:

- **Sample-Mounting Station**
The sample-mounting station is used to mount samples at the correct location in the sample holder. For more information about mounting samples, see Figure 5-8 and Section 3.3.
- **Sample Holders**
Sample holders are used to hold samples (see Figure 5-8); the holders screw onto the bottom of the sample rod. Quantum Design has provided five paddle-shaped sample holders.
- **Calibration Sample**
A palladium and Er:YAG calibration sample is used to calibrate the ACMS II option and to verify its accuracy.
- **Coilset Assembly**
The ACMS II coilset assembly is shipped as part of the ACMS II Option User's Kit.

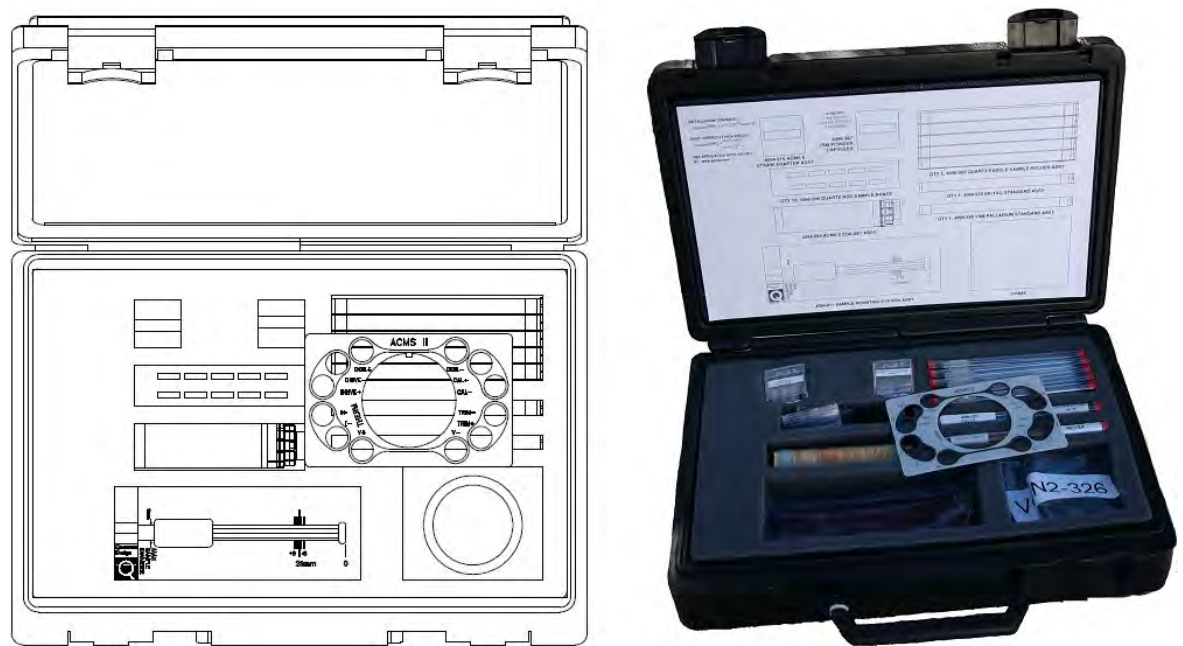


Figure 5-7. ACMS II Option User's Kit (4084-810)

5.4 Sample-Mounting Station

The sample-mounting station (4084-811) is used to precisely locate and measure (within 0.5 mm) the sample offset, which is the distance between the sample and the bottom of the sample holder (see Figure 5-8).

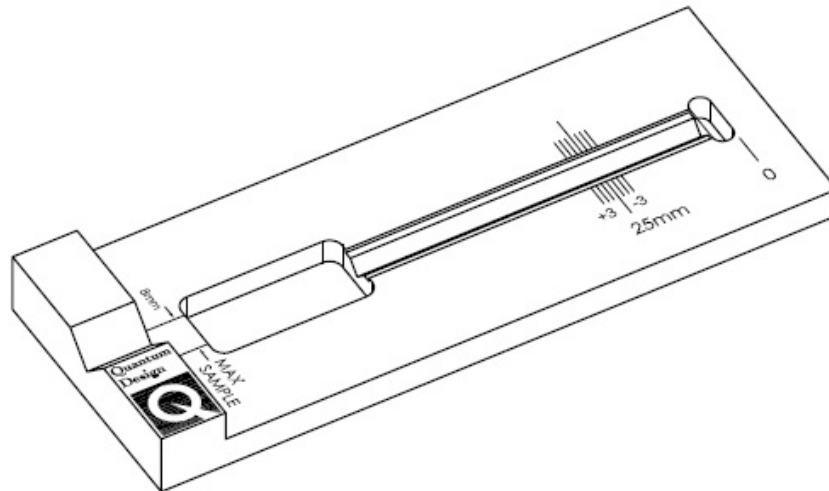


Figure 5-8. Sample-mounting station (4084-811)

Both AC and DC measurement algorithms requires an accurate measurement of this distance so that it can perform touchdown operations (See Chapter 4 for more details about touchdown operations).

5.5 ACMS II Electronics

5.5.1 Signal Cable Assembly

Figure 5-9 shows the preamp cable assembly (3084-801), which is the electrical connection between the coilset puck and the “SIGNAL” port (JM-1) on the Model CM-M ACMS II module. The “Grey Lemo” end of the cable is plugged into the gray connector at the PPMS probe head.

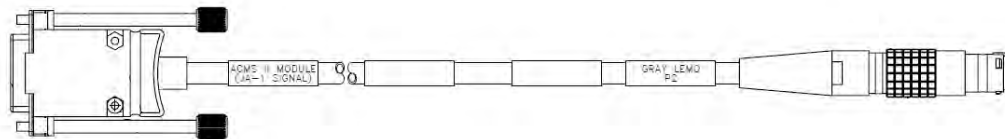


Figure 5-9. ACMS II signal/drive cable assembly (3084-801)

5.5.2 Motor Drive Cable

Figure 5-10 shows the DC motor drive cable (3096-200), which connects the back of the linear motor transport to the “SERVO” port (JA-1) on the Model CM-A (motor module). The drive cable provides drive-coil power to the linear motor transport, position-encoder read-back to the module, and serial communication between the linear motor transport and the module for diagnostic and configuration purposes.

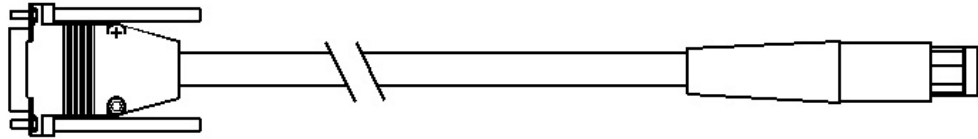


Figure 5-10. Motor drive cable (3096-200)

5.5.3 Model CM-A (Motor Module)

Figure 5-11 shows the Model CM-A motor module (4101-100), which provides all the power and logic that are necessary to drive the sample linear motor transport. All configuration and control of this module is through the ACMS II application software on the computer (PC) via the CAN-bus connector on the back panel of the module.

See Appendix A for a detailed description of the functions of this module.

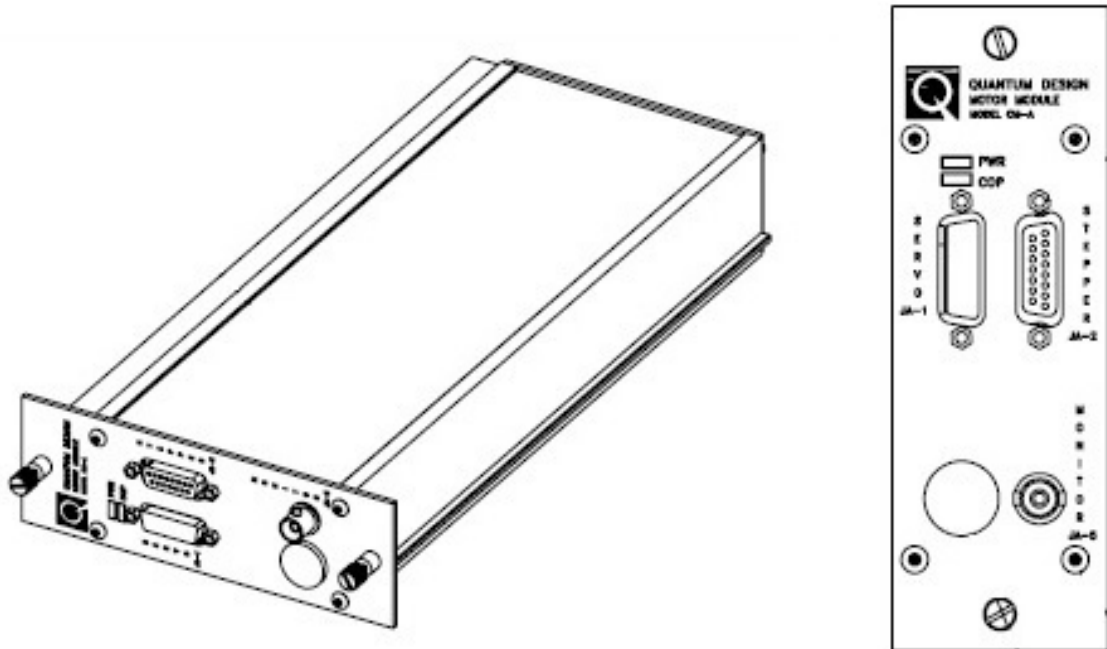


Figure 5-11. Model CM-A motor module (4101-100)

5.5.4 Model CM-M (ACMS II Module)

Figure 5-12 shows the Model CM-M (4101-430), which performs the synchronous detection (or “lock-in”) operation for the ACMS II option. The module provides the AC drive current for AC measurements, performs the signal detection for both AC and DC measurements, and measures the temperature of the ACMS II coil set. All configuration and control of this module is handled through the ACMS II application software on the computer (PC) via the CAN-bus connector on the back panel of the module.

See Appendix B for a detailed description of the functions of this module.

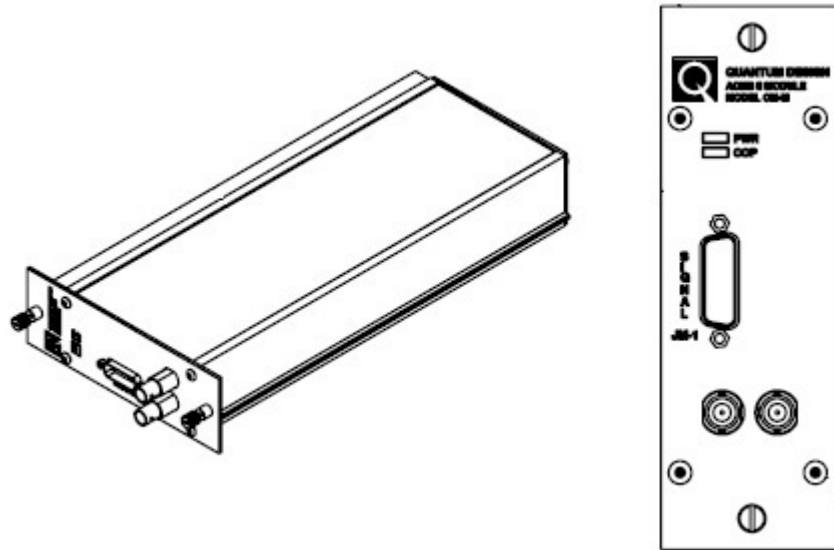


Figure 5-12. Model CM-M ACMS II module (4101-430)

5.5.5 Model 1000 (Modular Control System) (PPMS ONLY)

The Model 1000 modular control system (4100-001) is a general-purpose chassis (Figure 5-13) that houses, cools, and provides power to both the Model CM-A DC motor module and the Model CM-M ACMS II detection module. The Model 1000 can accommodate up to four additional modules for other PPMS options. The backplane provides connections for power as well as a CAN-based network connection to the computer (PC).

See the *Model 1000 Modular Control System User's Manual* for more information about the Model 1000 modular control system.

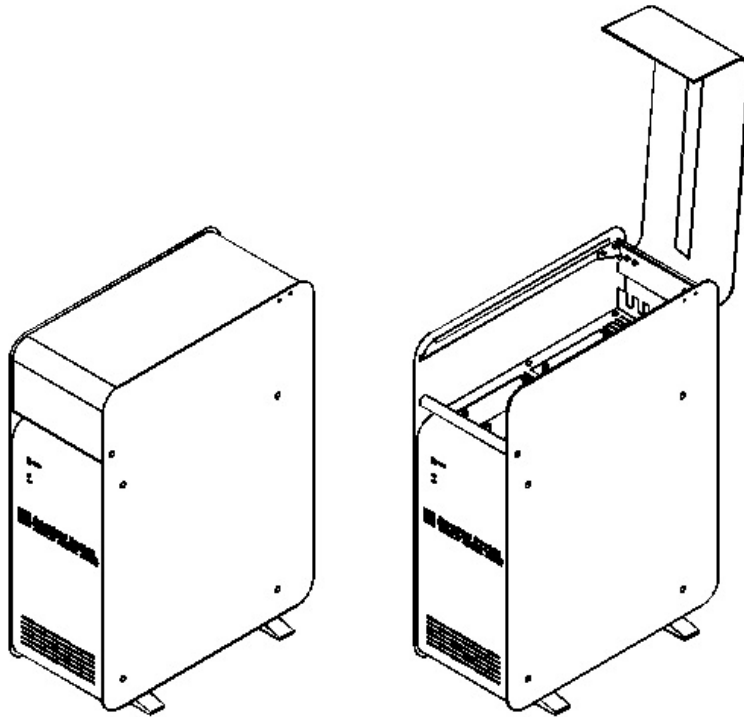


Figure 5-13. Model 1000 modular control system (4100-001)

5.5.6 CAN Computer Interface Kit

The CAN computer interface (or network adapter) kit contains the CAN-based network adapter, cable (3100-024), and software that are needed to connect the Model 1000 to the computer (PC).

For measurement platforms such as DynaCool and VersaLab, the CAN Computer Interface is built into the instrument.

ACMS II Software

6.1 Introduction

This chapter contains the following information:

- Section 6.2 describes option activation and deactivation.
- Section 6.3 describes the ACMS II-specific dialogs and menu items in the MultiVu dropdown menus.
- Section 6.4 describes the **ACMS II Control Center** and its components.
- Section 6.5 briefly describes sequence mode commands.
- Section 6.6 explains use of the ACMS II sequence-mode **Center Sample** command.
- Section 6.7 describes the format of ACMS II data files.

6.2 Activating and Deactivating the ACMS II Option

1. To activate the ACMS II application and open the **ACMS II Control Center**, select **Utilities >> Activate Option** from the dropdown menu of the MultiVu window (Figure 6-1).

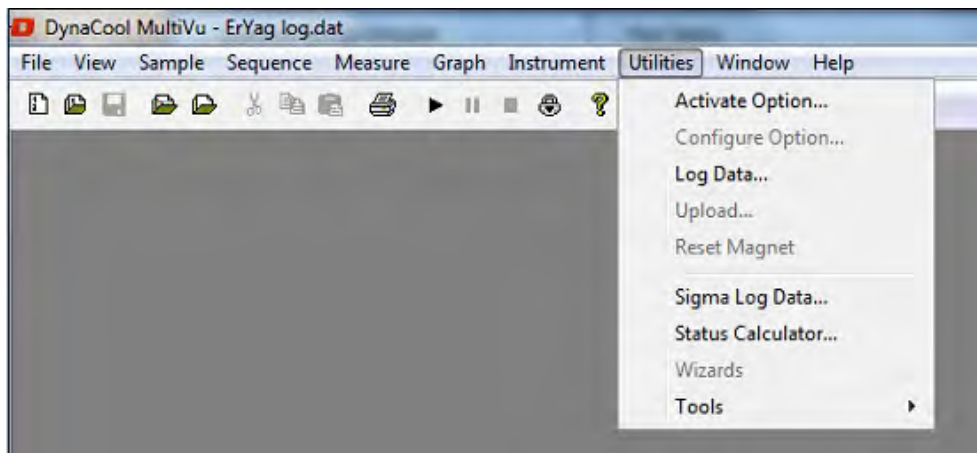


Figure 6-1. Using the MultiVu dropdown Utilities menu and Option Manager to activate the ACMS II Option

2. When the **Option Manager** dialog opens, select **ACMS II** and click on the **Activate** button. This will move the ACMS II option from the **Available Options** section of the dialog to the **Active Options** section. The **ACMS II Control Center** and the **ACMS II Log** window will open concurrently. When running in simulation mode, the **ACMS II Control Center** is titled **ACMS II SIM** (see Figure 6-2). To deactivate the ACMS II option, select it on the right side and click **Deactivate** in this dialog.

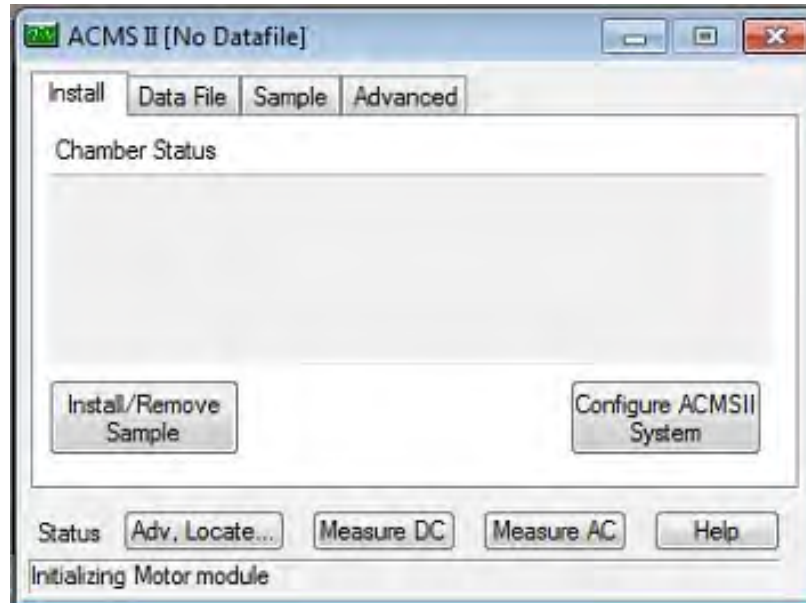


Figure 6-2. The MultiVu window and the ACMS II Control Center (ACMS II SIM), including the Install tab, the ACMS II Status area, and the MultiVu Status bar. Note that the ACMS II Log window has been minimized.

6.3 ACMS II Dropdown Menus

ACMS II-specific actions, dialogs, and commands are incorporated into the **View**, **Sample**, and **Measure** dropdown menus in the MultiVu window after you have activated the option. The other dropdown menus in the MultiVu window contain menu items that are common to all Quantum Design systems.

6.3.1 View

You can use the **View** dropdown menu at the top of the MultiVu window (Figure 6-3) to open ACMS II-specific dialogs as well as to perform general actions, such as opening and closing the MultiVu **Status Bar**, the sequence **Control Center** panel at the side of the window, or the tool bar.

- Select **ACMS II Status Log** to view the events logged during the current ACMS II activation. The file located in \ACMS II\Logfiles\ACMS IIlog.txt contains the entire history of this log and is a useful resource for troubleshooting.
- Select **ACMS II Error Count** to open a dialog showing only errors encountered as well as the total count.

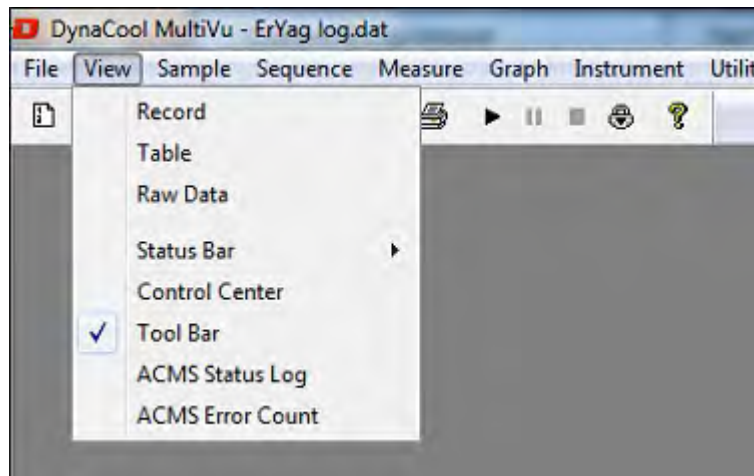


Figure 6-3. The MultiVu window and the View dropdown menu showing ACMS II immediate-mode commands

6.3.2 Sample

You can use the **Sample** dropdown menu at the top of the MultiVu window (Figure 6-4) to install and remove samples and to center the sample manually.

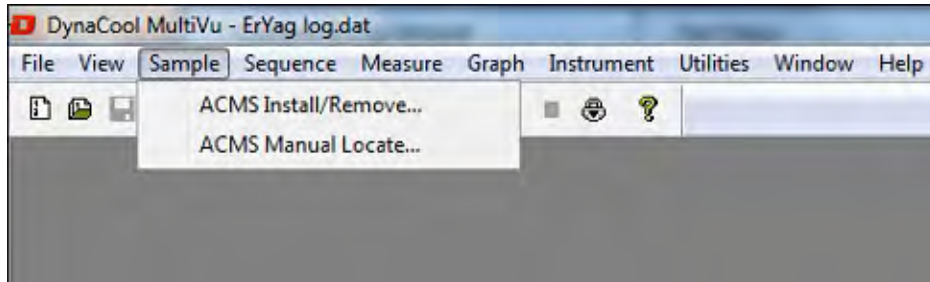


Figure 6-4. The MultiVu window and the Sample dropdown menu showing ACMS II immediate-mode commands

- To install a sample, designate an output data file, and center the sample, select **Sample >> ACMS II Install/Remove**. This selection opens the **ACMS II Install/Remove Sample Wizard** (Figure 6-5).
- To provide the software with the location of the sample, select **Sample >> ACMS Manual Locate**. The **Specify Sample Location** dialog will open so that you can enter the sample offset or perform an automatic scan. This dialog also appears when you use the **ACMS II Install/Remove Sample Wizard** to install a sample (Section 4.3.3).

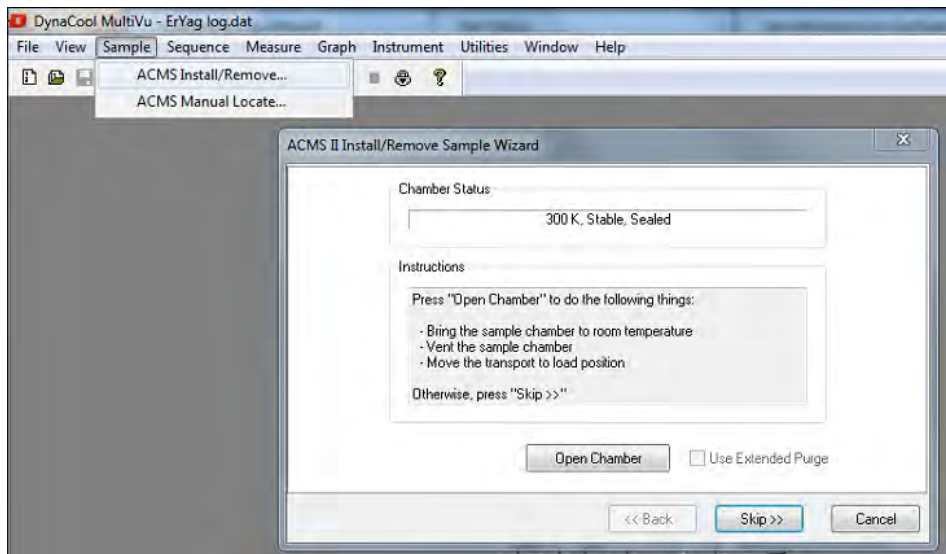


Figure 6-5. The Specify Sample Location dialog that opens from the dropdown menu when you select Sample >> ACMS II Manual Locate

6.3.3 AC Measure

If a sample has been installed in the ACMS II, you can use the **Measure** dropdown menu at the top of the MultiVu window (Figure 6-6) to open immediate-mode ACMS II measurement dialogs (e.g., for setting up and taking measurements, for centering the sample, and for adding a comment to an output data file). In the event you attempt to use commands in the **Measure** dropdown menu but you have not installed a sample, a popup message will open, directing you to first install and center a sample.



Figure 6-6. ACMS II immediate-mode commands in the context of the MultiVu window and the Measure dropdown menu

- To set up and take measurements, select **Measure** and choose either **AC Susceptibility** or **DC Magnetization** from the drop-down menu. The **ACMS II Measurement** dialog will open (Figure 6-6), with three tabs (or pages) of settings you can use to delineate your measurements. The same dialog appears when you click on the **Measure** button in the **ACMS II Control Center**. For a complete explanation of the **ACMS II Measurement** dialog, see Section 6.4.2.
- To center a sample, select **Measure >> ACMS II Center Sample**. This selection sequence opens the **ACMS II Center Sample** popup (Figure 6-7), which allows you to initiate touchdowns at will. As explained in Chapter 4, centering operations help ensure the accuracy of measurements.
- To add a comment to a ACMS data file, select **Measure >> ACMS II Datafile Comment**. When the **ACMS II Datafile Comment** popup appears (Figure 6-8), enter your comment in the text box. The text will be appended to the currently open ACMS II data file in the Comment column (first column).



Figure 6-7. The immediate-mode AC Center Sample popup



Figure 6-8. The immediate-mode AC Datafile Comment popup

6.4 ACMS II Control Center

The **ACMS II Control Center** opens within the MultiVu window as soon as you activate the ACMS II option and application software. Figure 6-9 shows the **ACMS II Control Center** and the commands, dialogs, tabs, buttons, and software prompts that organize measurement-related activities, making it easy for you to perform basic operations (e.g., create data files, install samples, and set up and initiate immediate-mode measurements).

6.4.1 ACMS II Control Center: Components

6.4.1.1 INSTALL TAB

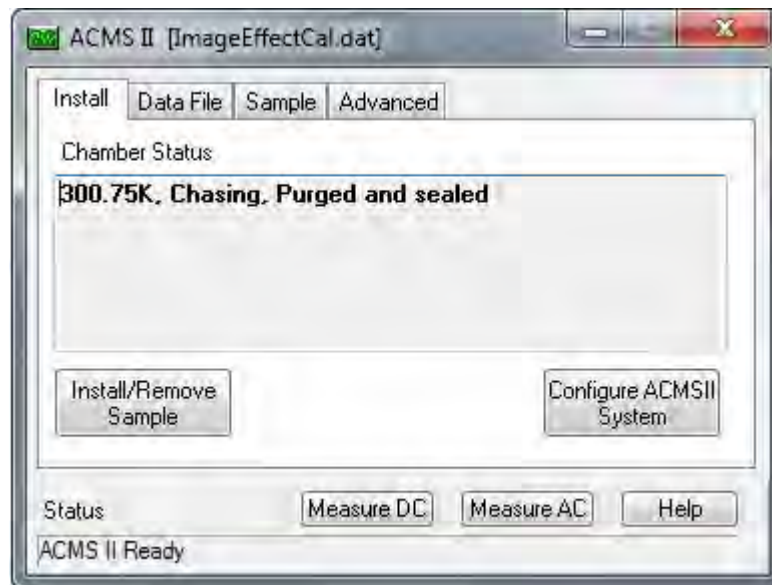


Figure 6-9. Install tab in the ACMS II Control Center

The **Install** tab provides access to two wizards:

- **Install/Remove Sample** guides the user through installation of a sample into the ACMS II measurement space and is outlined in Section 4.3.3.
- **Configure ACMS II System** will allow the user to select a different active ACMS II coilset (see Section 2.2.8). Coilsets are distinguished by serialization on the puck base (see Section 5.2.3) and there is a corresponding calibration file ACMSxxx.CFG in the ACMS\Calibration folder. One sign that an incorrect coilset is in use is that the temperature between coilset and base system differ by >1% or the ACMS option declares that the option thermometer is not responding.

6.4.1.2 DATA FILE TAB

The Data File tab (Figure 6-10) identifies the output data file that will contain the measurement data. If you have not selected an output data file, the File Name and Title panels in the Data File tab will be blank.

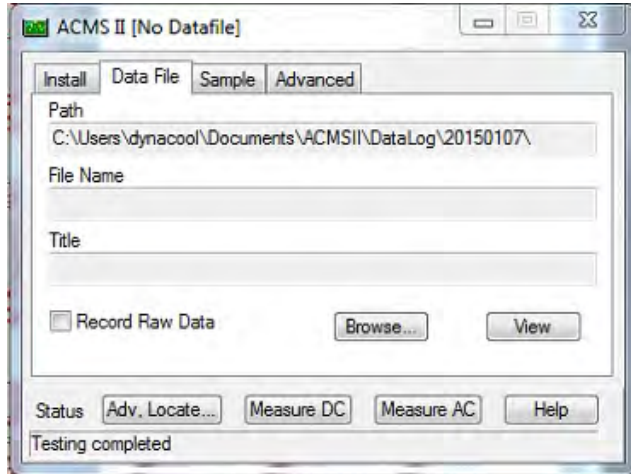


Figure 6-10. Data File tab in the ACMS II Control Center

View Button

The **View** button opens the graph view of the active output data file. To use the **View** button, you must have designated an output data file.

Browse... Button

The **Browse...** button initiates a series of dialogs that guide you through the process of designating (selecting or creating) a data file (see Fig. 6-11). The file-designation process and the dialogs are similar to the ones in the **Install/Remove Sample Wizard** (see Section 4.3.3).

Important: You must designate an output data file (by selecting an old file or by making a new one) if you want the measurement data saved. If no data file is designated, the measurement data will be written to the file `\Data\ACMS IIdefault.DAT`.

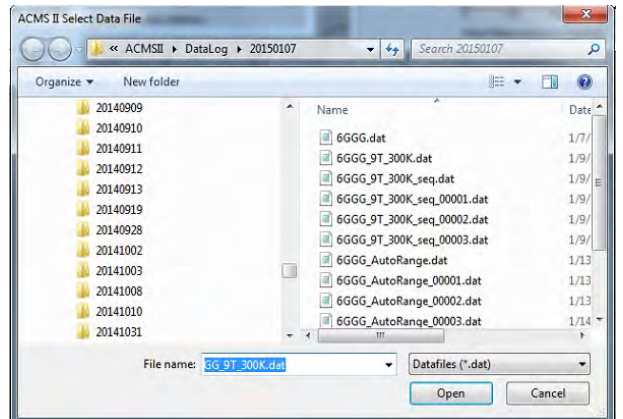


Figure 6-11. Browse button initiates a dialog to select data file

Creating a new data file will prompt the user to enter sample properties as shown in Figure 6-12. This information, recorded in the header of the data file, is for information only and is not used in calculating the reported magnetic moment.

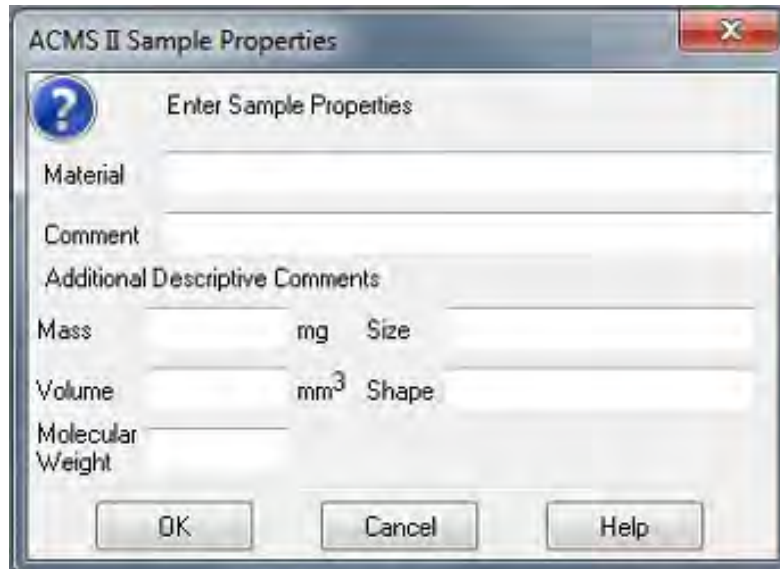


Figure 6-12. Recording sample properties

6.4.1.3 SAMPLE TAB

The **Sample** tab (Figure 6-13) is a read-only status display that shows the sample properties (e.g., material, comments, mass, and volume) for the active output data file. The information in the **Sample** tab originates from your entries in the **ACMS II Sample Properties** dialog, which are stored in the header of the output data file. If you select a pre-existing file to which you want data appended and bring the **Sample** tab forward, the information in the **Sample** dialog will reflect the entries in the header of the pre-existing file.

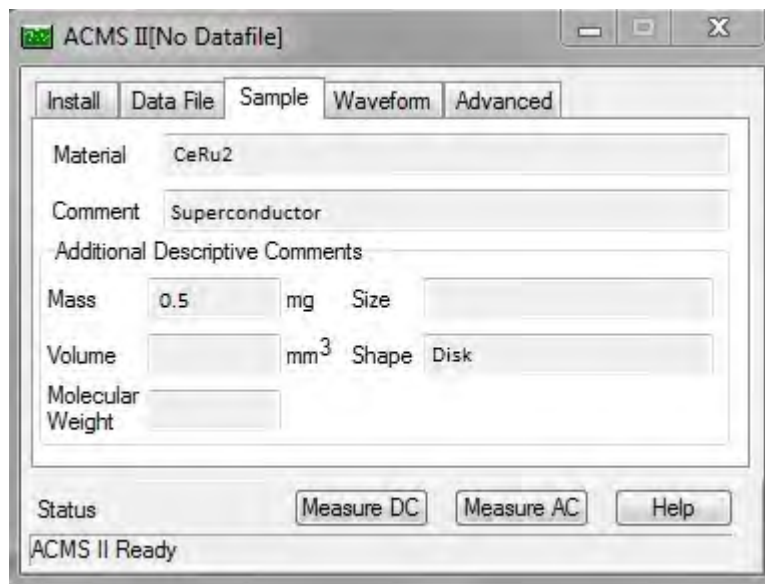


Figure 6-13. Sample tab in the ACMS II Control Center.

6.4.1.4 ADVANCED TAB

The **Advanced** tab (Figure 6-14) organizes support for troubleshooting, for example, checking calibrations and troubleshooting system performance. Hence, you will use the options on the **Advanced** tab only if you are an experienced ACMS II operator.

- The **Motor Friction Scan** button moves the sample linear motor transport through the full range of motion. The system plots the motor force (as current) as a function of position for both lifting the sample and lowering the sample. A difference between these two curves indicates friction due to ice or other obstructions.

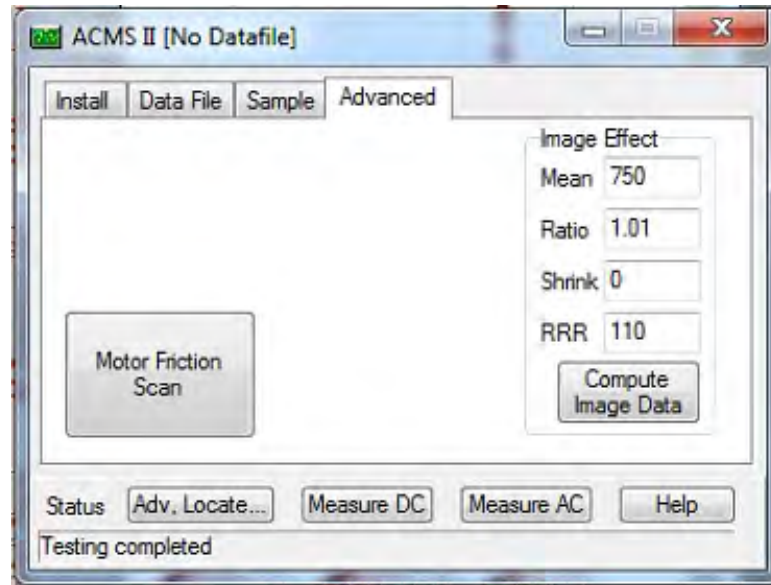


Figure 6-14. Advanced tab in the ACMS II Control Center

6.4.2 ACMS II Control Center: "Measure" Buttons

The **Measure DC** or **Measure AC** buttons in the **ACMS II Control Center** opens either the **ACMS II DC Magnetization** or the **ACMS II AC Susceptibility** measure dialog boxes (DC left and AC right of Figure 6-15), which organizes immediate-mode measurement settings and initiates measurements. The dialog has a section showing the measurements, three tabs (**Settings**, **Centering**, and **Advanced**), and four buttons (**Start (Stop)**, **Pause (Resume)**, **Close**, and **Help**).

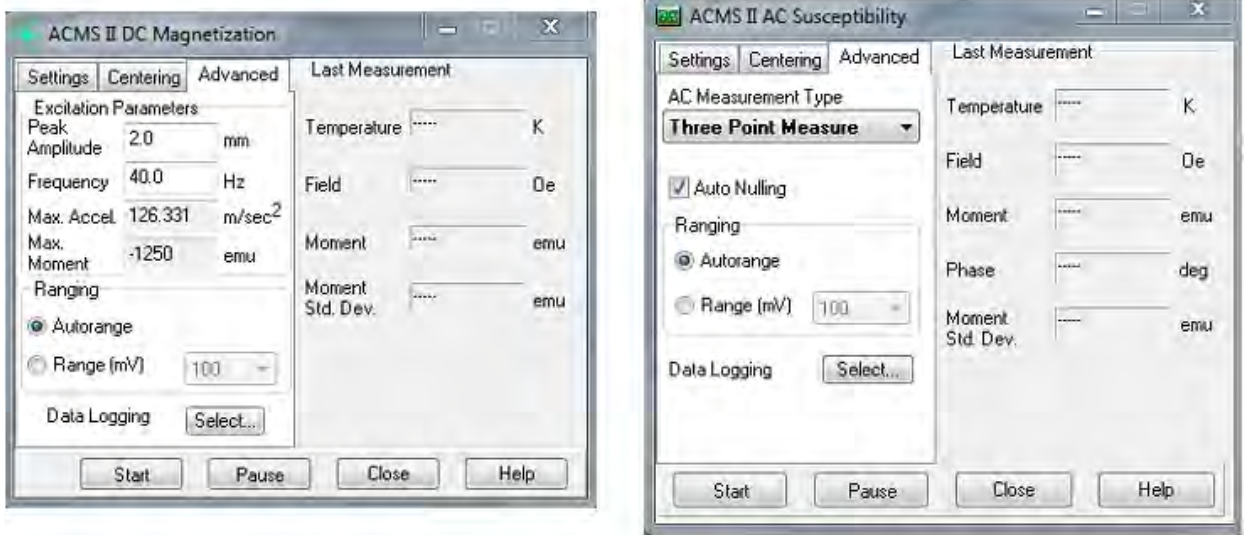


Figure 6-15. DC Magnetization and AC Susceptibility measure windows

6.4.2.1 MEASURE DIALOGS: BUTTONS

- The **Start (Stop)** toggle button starts and stops the measurement process, but it does not close the dialog.
- The **Pause (Resume)** toggle button only stops the system from performing measurements, as the system continues to oscillate the sample and execute any scheduled automatic touchdown operations. To restart the data output after a **Pause**, click on the **Resume (Pause)** toggle button.
- The **Close** button closes the **ACMS II DC Magnetization** or **ACMS II AC Susceptibility** measurement dialogs, but it does not stop the measurement.
- The **Help** button initiates the online Help menu.

6.4.2.2 MEASURE DIALOGS: LAST MEASUREMENT

The **ACMS II AC Susceptibility** and **DC Magnetization** measure dialogs are divided into two main areas: On the left side are the tabs with the measurement settings. On the right side is a **Last Measurement** area that displays the most recent **Temperature, Field, Moment, and Moment Std. Error** data, which are written to the open data file.

- **Temperature** represents the average temperature during the measurement, in Kelvin.
- **Field** represents the average field, in oersted.
- **Moment** represents the average of the moment over the averaging time, in emu.
- **Moment Std. Error** represents the error on the mean, that is, the error bar on the reported moment.

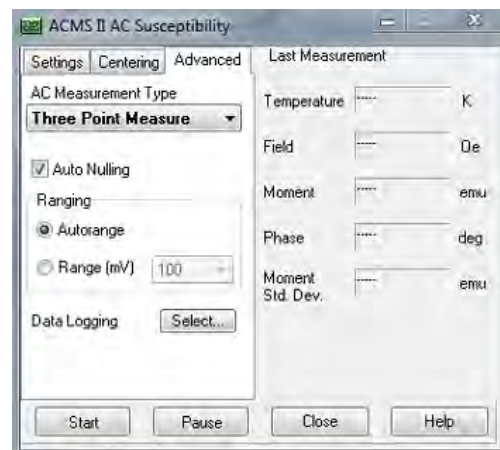


Figure 6-16. ACMS II AC Susceptibility measure dialog.

6.4.2.3 MEASURE DIALOGS: ADVANCED TAB

The **Advanced** tab of the **ACMS II DC Magnetization** measurement dialog (Figure 6-15) contains settings for **Excitation Parameters, Ranging, and PPMS Data Logging.**

The **Advanced** tab of the **ACMS II AC Susceptibility** measurement dialog (Figure 6-15, 6-16) contains settings for **AC Measurement, Ranging, and PPMS Data Logging.**

A short description for each of these settings is given below:

Excitation Parameters (DC magnetization dialog only)

The excitation parameters are explained by the equation:

$$\text{Position}(\text{time}) = [\text{Peak Amplitude}] \times \sin(2\pi * [\text{Frequency}] \times \text{time})$$

- The **Peak Amplitude** is typically set to 2 mm and can be varied from 0.1 to 5 mm with a recommended range of 0.5 to 4 mm. Low amplitudes allow for measuring a larger moment (see **Max Moment** value in this dialog) because the induced coil set signal is proportional to the amplitude. However, low amplitudes (<0.5mm) can result in an inaccurate reported moment. High amplitudes provide more coil set signal but also produce large accelerations (see **Max Accel.** value in this dialog) which can lead to higher noise in measurements.
Important: Quantum Design staff recommend that you limit the maximum **Peak Amplitude** to 4 mm, because the motor module could overheat at greater amplitudes. By limiting the maximum **Peak Amplitude** to 4 mm, you also ensure that the sample holder clears the puck surface. For example, taking into account that the detection coils are 40 mm above the puck surface and using a sample offset of 35 mm, the use of a **Peak Amplitude** of 5 mm or greater would cause the sample holder to touch the puck surface, which could dislodge the sample or the sample rod.
- The **Frequency** is typically 40 Hz; it specifies the frequency with which the linear motor oscillates the sample. It can be moved to a different value if there is interference at 40 Hz. See PPMS Service Note 1096-304 at www.qdusa.com for information about changing the vibration frequency.
- **Max. Accel.** is computed from **Frequency** (40 Hz) and the **Peak Amplitude** entry; it represents the maximum acceleration the sample will experience during a measurement in units of meter per second squared.

Important: Do not proceed with a measurement if your sample cannot tolerate accelerations of this magnitude. To reduce the acceleration, reduce the amplitude.

- **Max. Moment** is computed from **Frequency** (40 Hz) and the **Peak Amplitude** entry; it represents the maximum sample moment that can be measured using these settings.

Important: Do not proceed with a measurement if the magnetic moment of your sample is larger than the calculated **Max. Moment**, as the system will not be able to complete the measurement. Larger moments can be measured by using relatively small values for amplitude.

AC Measurement Type (AC susceptibility dialog only)

The **AC Measurement Type** pull down menu allows the user to select between: 1) Five point, 2) Three point and 3) One point measurements. See Table 4.1 in Section 4.4.1.1 for detailed descriptions of these measurement modes.

Auto Nulling

Checking the **Auto Nulling** box will initiate a nulling operation every time an AC measurement with a different excitation field frequency is performed. In general, nulling will result in quieter and more sensitive measurements.

Ranging

The **Ranging** setting refers to the way the system chooses the gain of the amplifiers in the ACMS II module during a measurement; the optimal setting is typically **Autorange**. The preamplifiers in the ACMS II module can change the gain ranges by factors of 10, depending on the size of the signal that is induced in the pickup coils. In the rare case when you need more control than is offered by **Autorange**, you can change the **Ranging** setting to a fixed value.

- **Autorange:** The system automatically increases the gain by a factor of 10 if the current peak signal drops below 2% of the current range. The system automatically decreases the gain by a factor of 10 if the current peak signal exceed 50% of the current range.
- **Fixed Range:** The system always uses the specified gain range. This can be useful when measuring samples which change signal rapidly (e.g., ferromagnets with a very sharp hysteresis loop) and range-changing is not desired.

Data Logging

In the **Data Logging** area of the Advanced tab of the **ACMS II AC Susceptibility** or **DC Magnetization** measure dialogs, the **Select ...** button opens the dialog (Figure 6-17), which lists additional system data items that you can choose to add to the current data file. These items are in addition to the ACMS II data items that are typically included, which are summarized in Table 6-1 (Section 6.8.2).

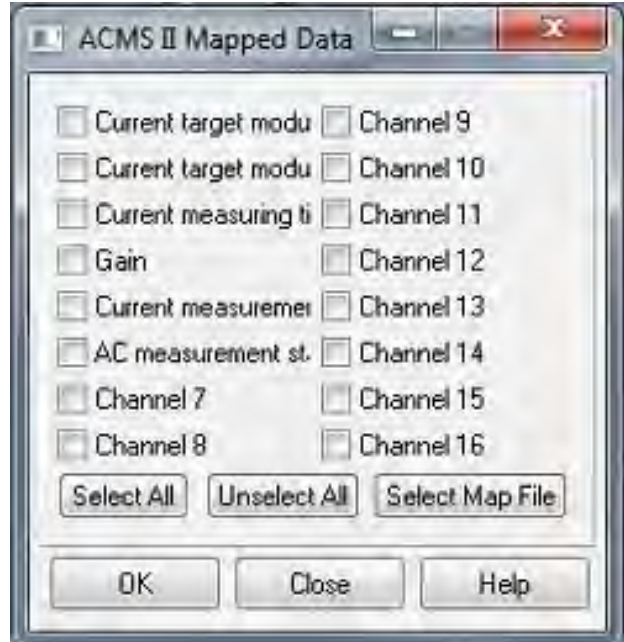


Figure 6-17. Data-logging dialog for selecting additional types of data to be collected

6.4.2.4 MEASURE DIALOGS: SETTINGS TAB

The **Settings** tab of the **ACMS II DC Magnetization** measurement dialog (Figure 6-18, left) contains settings for **Measure Type** and **Measurement Parameters**.

The **Settings** tab of the **ACMS II AC Susceptibility** measurement dialog (Figure 6-18, right) contains settings for **Measure Type**, and **Measurement Parameters**.

A short description for each of these settings is given below:

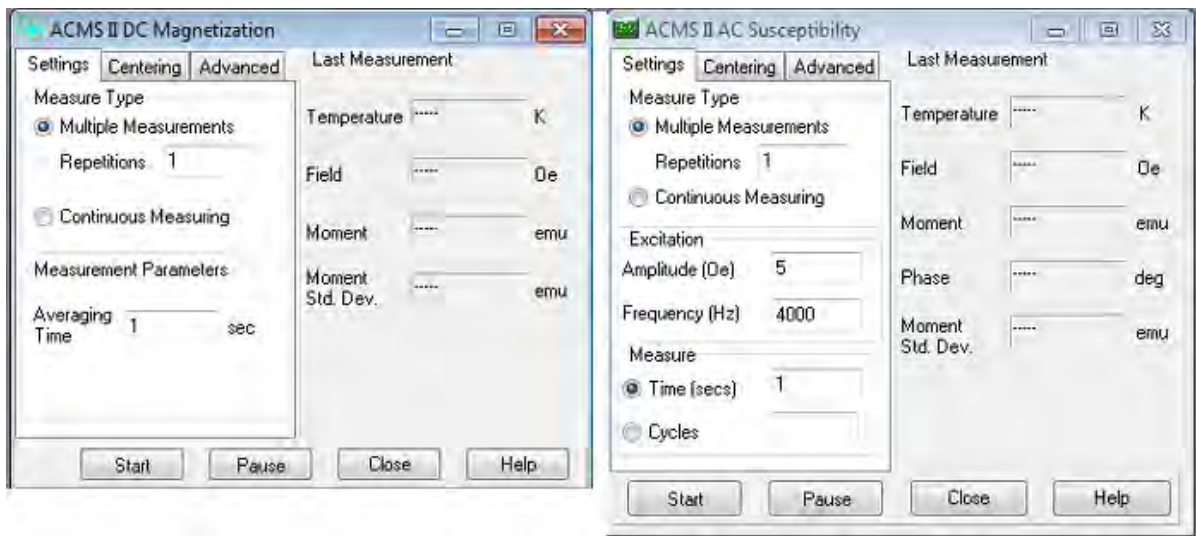


Figure 6-18. Settings tab in the Measure Dialogs.

Measure Type

The **Multiple Measurements** mode collects the number of data points specified in the “Repetitions” field upon pressing the **Start** button. In addition, measurements can also be taken in **Continuous** mode in which new data is collected and written to the data file until the **Stop** button is pressed.

Excitation Parameters (AC susceptibility dialog only)

The **Excitation Parameters** are specified by the user to define the Amplitude (Oe) and Frequency (Hz) of the magnetic excitation field during AC Susceptibility measurements.

Measurement Parameters (DC Magnetization only)

- **Averaging Time (secs)** specifies the boxcar average duration for each measurement and will be an integer number of periods of sample oscillation. A practical minimum for this time interval is 0.5 seconds and a typical value is 1 second.

Measure (AC Susceptibility only)

- **Time (secs)** specifies the boxcar average duration for each measurement and will be an integer number of periods of sine wave excitation. A practical minimum for this time interval is 0.5 seconds and a typical value is 1 second.
- **Cycles** is the number of sine wave excitation periods that elapses between recorded measurements.

6.4.2.5 MEASURE DIALOGS: CENTERING TAB

The **Centering** tab displays the automatic centering settings that are in effect (see Figure 6-19). Normally, you will not need to adjust these settings.

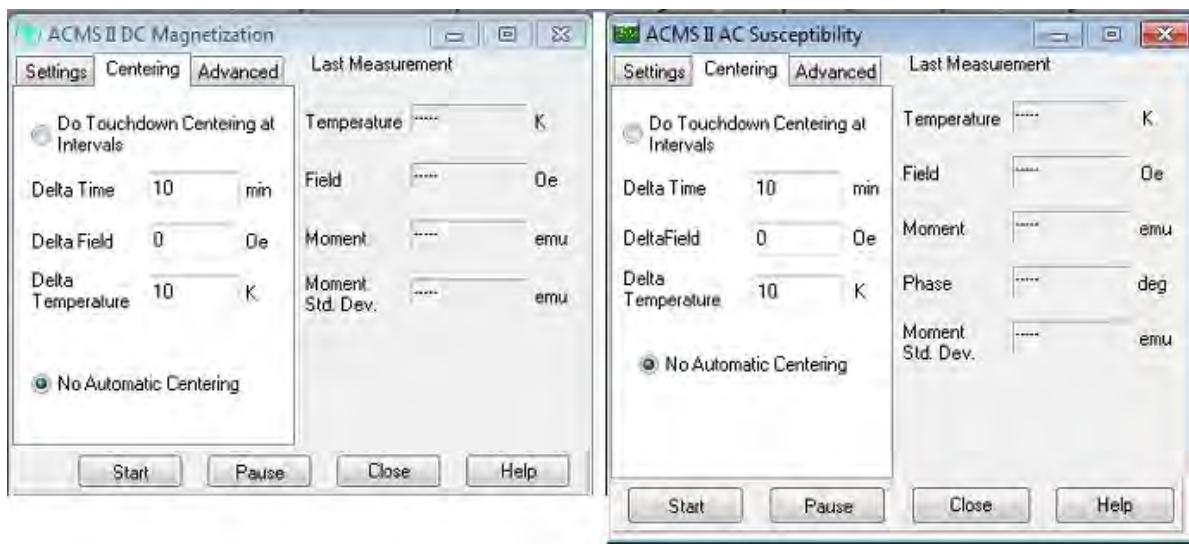


Figure 6-19. Centering tab in the Measure Dialogs.

It is recommended to use touchdown centering to ensure the sample remains centered in the coil set.

Touchdown operations will occur when any one of the monitors (time, field or temperature) has been exceeded. The default parameters of Delta Time = 10 minutes and Delta Temperature = 10 K will be sufficient for most cases. Selecting zero (0) for any of the monitors will turn it off. For instance, Delta Field = 0 should be used for all systems except the 16 tesla PPMS (it is provided because the stray field of the main magnet can slightly pull on the motor and change the operating height of the sample).

Touchdowns result in an approximately 20 second interruption in measurements. If this is unacceptable in your measurements then centering can be triggered explicitly by selecting **Measure >> ACMS II Center Sample** or using the **Center Sample** sequence command.

6.5 Overview of Sequence-Mode ACMS II Commands

ACMS II sequence-mode commands are, essentially, encapsulated versions of ACMS II immediate-mode measurement commands. The ACMS II sequence-mode commands have interactive dialogs that help you specify your measurements, and these dialogs are similar to the ones used to set up the immediate-mode measurements.

6.5.1 Taking an AC Susceptibility Measurement in Sequence Mode

To take a measurement in sequence mode, you first define the sequence file and include the measurement command in the sequence file. Then after you install the sample, you run the sequence, and the measurement is executed when the measurement command is read in the running sequence. A single sequence file may include any number of measurement sequence commands.

The **AC Susceptibility** measurement sequence command can either take a single AC measurement—just like the immediate-mode AC measurement—or it can function like a scan sequence command and take multiple measurements within a control loop. **AC Susceptibility** functions like a scan command if you define an amplitude or frequency range for the command parameters. Refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual* for detailed information about scan commands and control loop operation.

Complete the following steps to take an AC measurement in sequence mode:

1. Select a new or existing sequence file.
2. Select the **AC Susceptibility** command, which is in the **Measurement Commands ACMS** group in the sequence command bar. Then select the **Excitation** tab in the **ACMS II Measure AC Susceptibility** dialog box. See figure 6-20 (center).

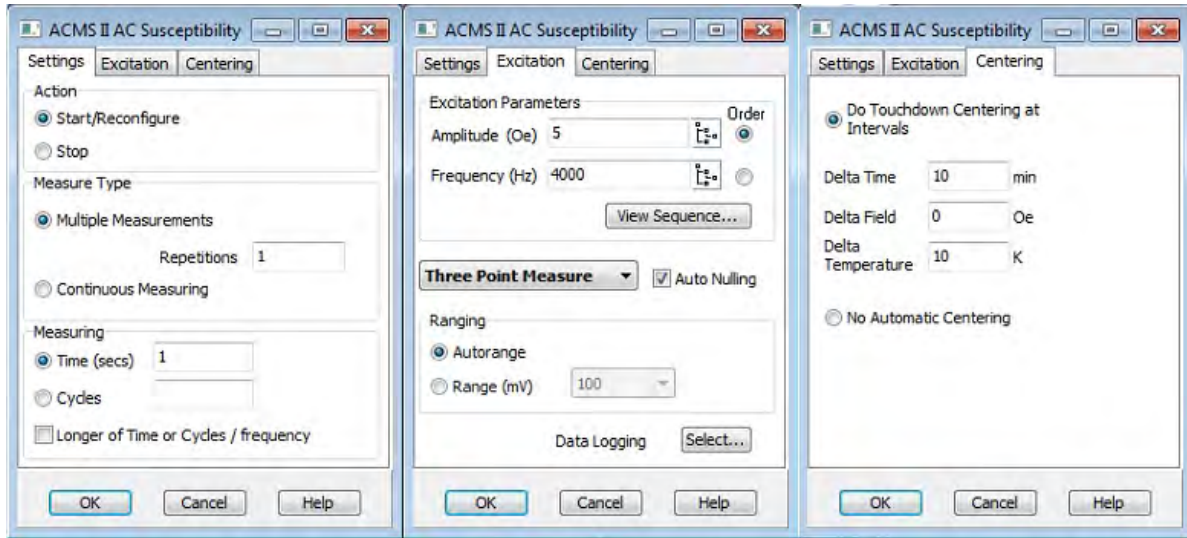


Figure 6-20. Tabs in **ACMS II AC Susceptibility Sequence Command Dialog Box**

- a. Under **Excitation Parameters** check the **Order** button that determines whether the amplitude or frequency is measured first. The measurement order may be important if you are using the **AC Susceptibility** command like a scan command.
- b. Define the **Amplitude** of the sine wave excitation that is applied to the sample. Do one of the following:
 - Enter a single value in the **Amplitude** text box.
 - Select the **Amplitude** button and define an amplitude range in the **Scan AC Field** dialog box. Defining a range prompts the **AC Magnetization** command to function like a scan command. Do the following: specify the initial and final set points, select a spacing mode (see table 6-1), specify the increment between the set points (if applicable), and specify the number of set points.

Table 6-1. Amplitude Spacing Modes

SPACING MODE	DEFINITION
Uniform	<i>Uniform spacing equally spaces the set points. This mode allows you to define the increment separating the set points. Uniform spacing is the default spacing mode.</i>
log(H)	log(H) spacing logarithmically spaces the set points, so defining the increment separating the set points is unnecessary.
H ²	<i>H² spacing equally spaces the set points in the square of the field, so defining the increment separating the set points is unnecessary.</i>

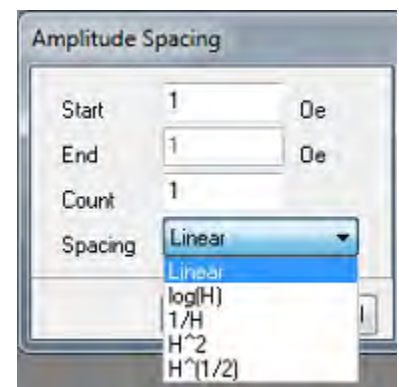


Figure 6-21. **Scan AC Field Dialog Box**



Not all amplitude values can be achieved in all frequency and temperature ranges. Amplitudes up to 10 Oe are producible at all frequencies and temperatures.

- c. Define the **Frequency** of the sample excitation signal. Do one of the following:
 - Enter a single value in the **Frequency** text box.
 - Select the **Frequency** button and define a frequency range in the **Scan Frequency** dialog box. Defining a range prompts the command to function like a scan command. Do the following: specify the initial and final set points, select a spacing mode (see table 6-2), specify the increment between the set points (if applicable), and specify the number of set points.

Table 6-2. Frequency Spacing Modes

SPACING MODE	DEFINITION
Uniform	<i>Uniform spacing equally spaces the set points. This mode allows you to define the increment separating the set points. Uniform spacing is the default spacing mode.</i>
log(f)	log(f) spacing logarithmically spaces the set points, so defining the increment separating the set points is unnecessary.
1/f	<i>1/f spacing uses the inverse frequency to equally space the set points, so defining the increment separating the set points is unnecessary.</i>

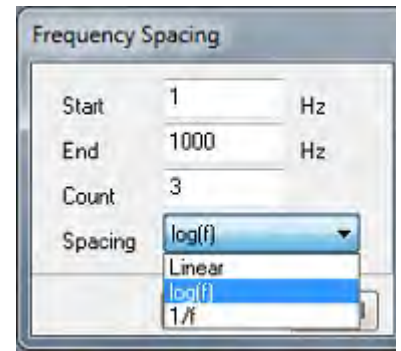


Figure 6-22. Scan Frequency Dialog Box

- d. Select the **View Sequence** button if you want to see a visual representation of the control loop measurement that is defined by the amplitude and frequency ranges. For example, the view tree in figure 6-23 depicts a control loop for a **Moment vs. Amplitude** primary scan mode measurement. Measurements are taken at multiple frequencies while the system holds a single amplitude.
- e. Select the type of measurement from the popup (see Table 4-1).

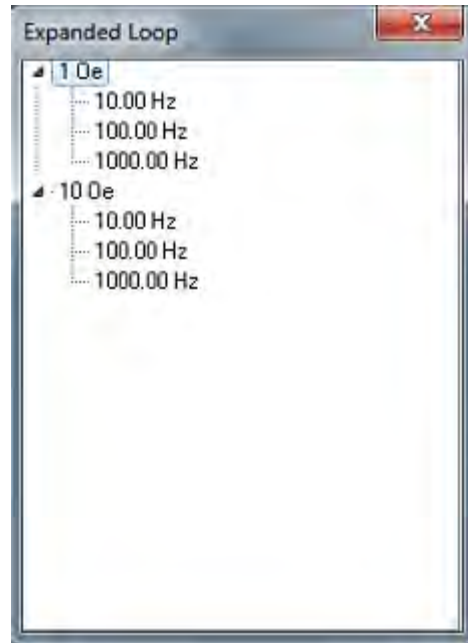


Figure 6-23. ACMS II View Tree

- f. Choose the **Ranging** mode used for voltage detection. **Autorange** changes the gain setting at the beginning of every measurement so that the optimum setting is selected. **Fixed Range** uses only a single user-specified gain setting.
3. Select the **button next to the “Data Logging” entry**, and then select the data items you want to save to the measurement data file. By default, the general system status, system temperature, magnetic field, and the sample temperatures are saved to the file.

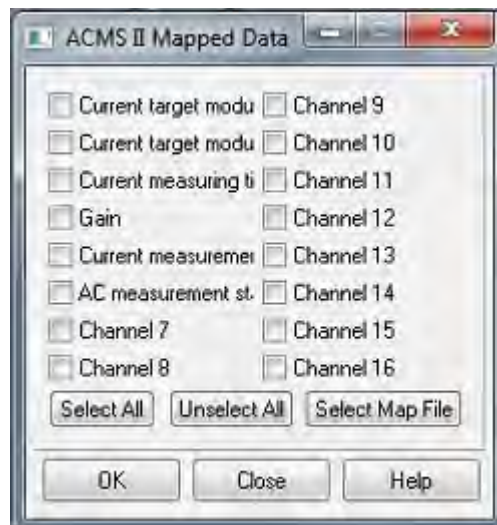


Figure 6-24. **Mapped Data** Tab in **AC Magnetization Sequence Command** Dialog Box

4. Switch to the “Settings” tab and define the averaging time.
5. Select the measurement type and action.
6. Select the **OK** button in the **AC Magnetization** dialog box to add the **AC Magnetization** command to the sequence.

7. Save the sequence.
8. Select a data file and install the sample just as you would for any immediate-mode measurement.

During the sequence run, you may also prompt the ACMS II software to save the sequence measurement data to different data files. Remember to save the sequence again if you make any additional changes to it.

Select any **Run Sequence** command in PPMS MultiVu. Then wait for the sequence run to finish. The commands in the sequence determine the length of time the sequence runs. When the run is complete, all **Run** commands are enabled. The measurement data is automatically saved to the data file you previously selected.

6.5.2 Taking a DC Magnetization Measurement in Sequence Mode

To take a measurement in sequence mode, you first define the sequence file and include the measurement command in the sequence file. Then after you install the sample, you run the sequence, and the measurement is executed when the measurement command is read in the running sequence. A single sequence file may include any number of measurement sequence commands.

Complete the following steps to take a DC measurement in sequence mode:

1. Select a new or existing sequence file.
2. Select the **DC Moment** command, which is in the **Measurement Commands ACMS** group in the sequence command bar. Then select the **Measure** tab in the **DC Magnetization** dialog box, and define the DC measurement as follows.
 - a. Define the number of **Scans** that are taken and averaged to obtain a measurement value.
 - b. Select the **Ranging** mode used for voltage detection. **Autorange** changes the gain setting at the beginning of every measurement so that the optimum setting is selected. **Fixed Range** uses only a single user-specified gain setting.

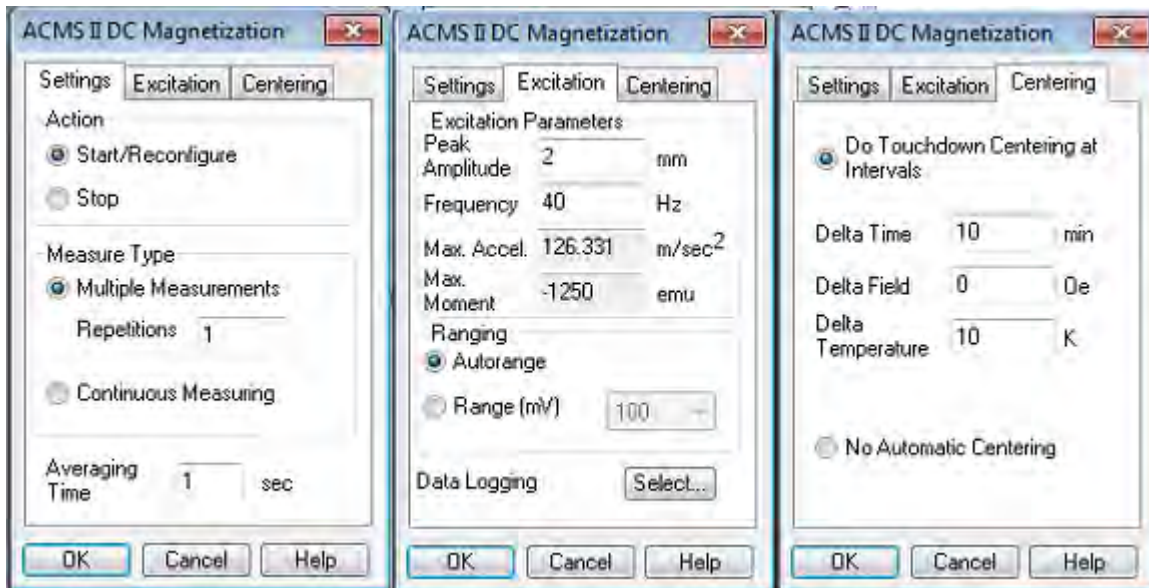


Figure 6-25. Settings Tab in DC Magnetization Sequence Command Dialog Box

3. Select the **button next to the "Data Logging" entry**, and then select the data items you want to save to the measurement data file. By default, the general system status, system temperature, magnetic field, and the sample temperatures are saved to the file.
4. Switch to the "Settings" tab and define the averaging time.
5. Select the measurement type and action.
6. Select the **OK** button in the **DC Magnetization** dialog box to add the **DC Moment** command to the sequence.
7. Save the sequence.
8. Select a data file and install the sample just as you would for any immediate-mode measurement.

During the sequence run, you may also prompt the ACMS II software to save the sequence measurement data to different data files. Refer to section 6.4.3. Remember to save the sequence again if you make any additional changes to it.

9. Select any **Run Sequence** command in PPMS MultiVu. Then wait for the sequence run to finish. The commands in the sequence determine the length of time the sequence runs. When the run is complete, all **Run** commands are enabled. The measurement data is automatically saved to the data file you previously selected.

6.6 Sequence-Mode ACMS II "Center Sample" Command

Center Sample sequence commands can be combined with non-ACMS II sequence commands and looping constructs. If you choose not to use automatic centering, you will need to perform centering operations by using the dropdown menu or by inserting centering operations into a

sequence file. As explained in Chapter 4, centering operations help ensure the accuracy of measurements, keeping the centering position stable to within about 0.1 mm at the center of the pickup coils by informing the system about sample position shifts with respect to the coilset.

To insert a centering operation into a sequence file, click on the **Center Sample** sequence command in the **Sequence Commands** bar. This opens the **ACMS II Center Sample Sequence** dialog shown in Figure 6-26.

When the sequence file is run and the program encounters the **Center Sample** sequence command, it temporarily halts the current **ACMS II Measure** command. During the pause, the program performs a centering operation and adjusts the center of oscillation for the measurement. Then it resumes the measure command, continuing from the point where it stopped. This operation takes about 20 seconds.



Figure 6-26. Sequence-mode ACMS II Center Sample command: ACMS II Center Sample Sequence popup

The **Center Sample** sequence command is invaluable when you have disabled automatic centering (by selecting **No Automatic Centering**), but it is also useful when automatic centering has been enabled. In the latter case, you can place the **Center Sample** command in your sequence just before measurements that you would like to have performed without the interruption of a touchdown operation. The measurement is still subject to the other parameters (e.g., **Averaging Time** and **Logging Interval**) that you have set, but by explicitly executing the **Center Sample** command, you reset the beginning of the interval.

6.7 ACMS II Data Files

Data files have a `.dat` file extension. To save ACMS II measurement data, you must open a measurement data file *before* you start the measurement. You designate data files by creating a new one or selecting a pre-existing one, as is explained in Section 6.4.1.3.

6.7.1 Data File Headers

The header of a data file contains information such as the title of the data set and the sample properties. You have the opportunity to include this information when you create the file—this is the only time you can add this information to the data-file header. Instead, after a file has been created, you can append comments.

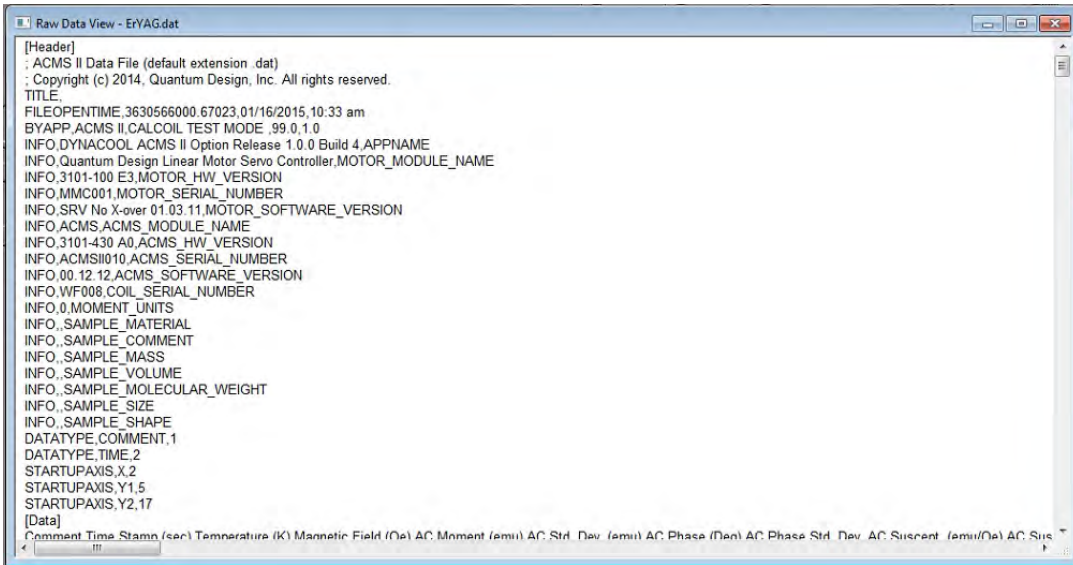


Figure 6-27. A Raw Data view of the ACMS II data file

6.7.2 Fields in ACMS II Data Files

Table 6-3 defines the fields in a ACMS II data file, and Section 6.4.1.3 explains how to create ACMS II measurement data files.

Table 6-3. Definitions of column headers for ACMS II data files (*.dat files), shown in the order they appear.

COLUMN HEADER/TERM	DEFINITION
Comment	user-specified comment; added by using the Datafile Comment command
Time stamp (sec)	time stamp of the measurement
Temperature (K)	average temperature (T) of the sample during measurements. The sample temperature is measured by the coil thermometer.
Magnetic field (Oe)	average magnetic field during measurement
AC Moment (emu)	Average AC magnetic moment of the sample
AC Std. Dev. (emu)	Standard deviation of the AC moment
AC Phase (Deg)	Phase angle of the AC moment in relation to the drive field
AC Phase Std. Dev. (Deg)	Standard deviation of the phase angle
AC Suscept. (emu/Oe)	Magnetic susceptibility of the sample (moment/drive)
AC Suscept. Std. Dev. (emu/Oe)	Standard deviation of the susceptibility
AC X' (emu/Oe)	In-phase component of the sample susceptibility
AC X'' (emu/Oe)	Quadrature component of the sample susceptibility
AC X' Std. Dev. (emu/Oe)	Standard deviation of the in-phase component
AC X'' Std. Dev. (emu/Oe)	Standard deviation of the quadrature component
AC Drive (Oe)	AC drive field applied to the sample
AC Frequency (Hz)	AC frequency of the drive field
DC Moment (emu)	Average DC magnetic moment of the sample (synchronous component)
DC Std. Err. (emu)	Standard error of the DC moment
DC Quad. Moment (emu)	Quadrature component of the DC moment (out of phase component)

COLUMN HEADER/TERM	DEFINITION
Range (mV)	Range 1mV, 10mV, 100mV of the signal amplifier
AC=1 DC=2 Locate=3	Measurement type as indicated by the number
Measure Type	AC measure type: 1=one point 3=three point 5=five point
Measure Count	Number of signal (V) readings used to calculate the moment
Measure Time (sec)	Elapsed time of the measurement
Measure Number	Sequential number for a measurement in a multi-measurement command
AC' Raw (emu/Oe)	In-phase component of the sample susceptibility without corrections
AC'' Raw (emu/Oe)	Quadrature component of the sample susceptibility without corrections
DC' Raw (emu)	Average DC magnetic moment (in-phase) of the sample without corrections
DC'' Raw (emu)	Quadrature component of the DC moment without corrections
Coil InPhase (V)	Average in-phase component of the raw signal voltage readings
Coil Quad. (V)	Average quadrature component of the raw signal voltage readings
Max Signal (V)	Maximum voltage read by the AD converter during the measurement (future)
Motor Frequency (Hz)	Measured vibration frequency of the motor used for the DC measurement
Motor Ampl. (mm)	Measured vibration amplitude of the motor used for the DC measurement
System temp. (K)	block temperature
System field (Oe)	currently the same as the "Magnetic Field (Oe)" column
Min. temperature (K)	minimum temperature reading of the coil thermometer for this measurement
Max. temperature (K)	maximum temperature reading of the coil thermometer for this measurement
Min. field (Oe)	minimum field reading of the coil thermometer for this measurement
Max. field (Oe)	maximum field reading of the coil thermometer for this measurement
Pressure (torr)	pressure in sample chamber
Temp. Status (code)	Status of the temperature of the system (eg. Stable, see system manual)
Field Status (code)	Status of the system magnet (eg. holding, see system manual)
Chamber Status (code)	Status of the chamber of the system (eg. Purged, see system manual)
ACMS II status (code)	status codes unique to the ACMS II module
Motor status (code)	status codes unique to the motor module
Measure status (code)	error condition codes of varying severity. Zero indicates no errors.
Center position (mm)	Average position of the transport for the current data point. Position is reported in motor coordinates and does not reflect the sample offset position that is shown in the locate dialog.
Transport action	1 = measurement 2 = auto-touchdown 3 = manual touchdown
Motor lag (deg)	phase lag between motor drive current and motion
Motor Current (Amps)	Motor current required to maintain position and amplitude
Motor Heatsink Temp. (C)	Temperature of the motor heatsink
Motor Board Temp. (C)	Temperature of the motor board

Model CM-A Sample Motor Module

A.1 Introduction

This appendix contains the following information:

- Section A.2 provides a functional overview of the Model CM-A sample motor module, including a block diagram and electrical specifications.
- Section A.3 describes the front panel and relevant components of the Model CM-A sample motor module.
- Section A.4 describes the back panel and relevant components of the Model CM-A sample motor module.
- Section A.5 describes maintenance of the Model 1000.

A.2 Functional Overview

The Model CM-A (4101-100) is a servomotor controller module that was designed with the specific needs of the sample motor in mind. Figure A-1 shows the module and the front panel.

The principle function of this module is to provide closed-loop servo control to a linear motor equipped with a position encoder output. A programmed wave table allows the module to drive the motor sinusoidally at 40 Hz. The servo loop is closed digitally at about 2000 Hz using a 16-bit current source and the read-back from the position encoder. For use with other synchronous detection hardware, including the Model CM-M ACMS II detection module, the real-time encoder position is output digitally, using a high-speed serial port, and as a voltage through a BNC connector. Other features include in-system programmable on-board flash memory for program storage and a serial ROM for calibration and other configuration data.

The module is designed to plug into the Model 1000 modular control system or an equivalent host chassis that can provide power and the required CAN network signals that communicate with the module.

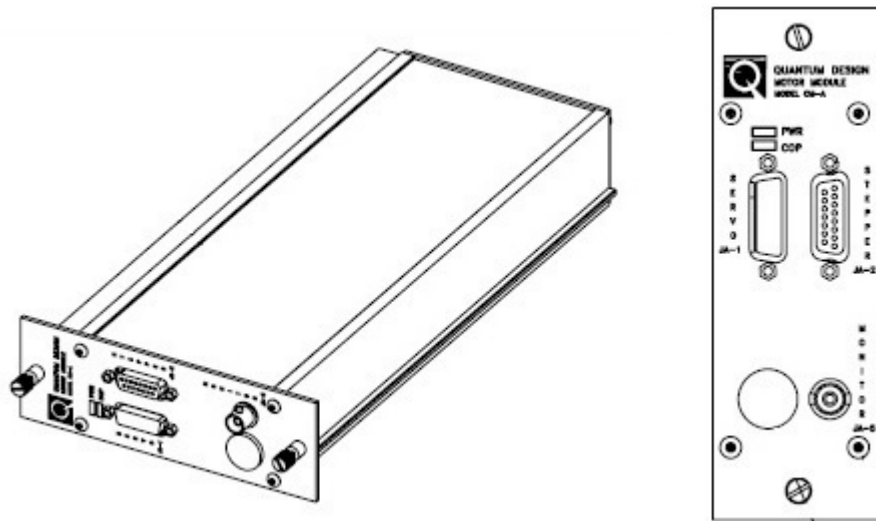


Figure A-1. Model CM-A sample motor module (4101-100)

A.2.1 Functional Block Diagram

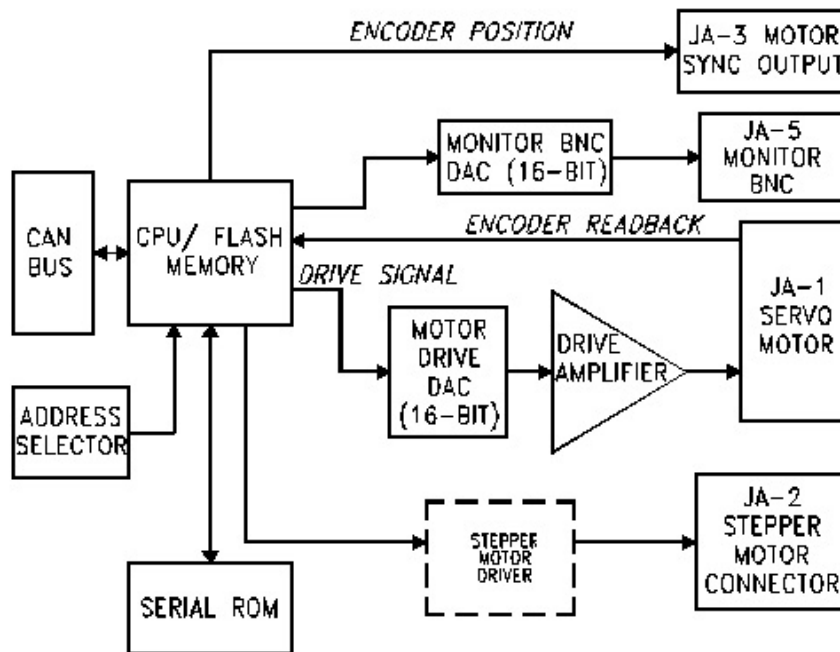


Figure A-2. Abridged functional block diagram of Model CM-A sample motor-module specifications

A.2.2 Specifications

Table A-1. Electrical specifications for Model CM-A motor module

Drive Current Limit	3 A
Drive Voltage Compliance Limit	20 V peak
Encoder Range	32-bit
Supply Voltage	±24 V DC

A.3 Model CM-A Sample Motor Module: Front Panel

A.3.1 Indicator LEDs

The front panel of the Model CM-A motor module has two LEDs in the top left, as shown in Figure A-1. The PWR LED indicates the power-on status of the module. The COP (CANopen Protocol) LED indicates the status of the CAN network controller. Table A-2 outlines the LED states and provides solutions in the event of a problem.

Important: The error information in Table A-2 refers to situations that persist for longer than about 15 seconds. Typically, when the module is powered on, the LEDs may briefly flash red before they turn green. This is a normal part of the startup or reset sequence.

Table A-2. LED guide for Model CM-A VSM motor module

LED	COLOR	STATUS	MEANING AND/OR SOLUTION
PWR	Green	On	The processor is running with no errors (normal).
	Red	Flashing	Errors were encountered during the self-test. The flashing sequence can be used to determine the cause of the failure.
COP	Green	On	CAN status is operational (normal).
		Flashing	CAN status is pre-operational. Verify that cable is connected to PC.
	Red	On or flashing	Error on the CAN bus. Contact Quantum Design for assistance.

If you are unable to achieve operation with both LEDs green, please contact Quantum Design for assistance.

A.3.2 Connectors and Pinout Tables

A.3.2.1 JA-1: SERVO CONNECTOR

This connector is used to provide the current drive to the motor and read back the position information from the encoder. This connector also supports serial communication to logic associated with the motor (e.g., serial ROM for storing calibration or configuration information about the motor).

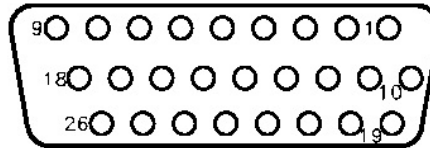


Figure A-3. JA-1: Servo connector for the Model CM-A motor module

Table A-3. JA-1: Servo connector for the Model CM-A motor module

PIN	FUNCTION
1	Motor -
2	+5 V
3	Encoder Input A+
4	Encoder Input B+
5	Unused Encoder Input Z+
6	n.c.
7	n.c.
8	Motor Max Limit Switch
9	Motor Min Limit Switch
10	Motor +
11	Ground
12	Encoder Input A-
13	Encoder Input B-
14	Unused Encoder Input Z-
15	n.c.
16	n.c.
17	Motor Max Limit Switch Rtn
18	Motor Min Limit Switch Rtn
19	Serial Com Data Out
20	Serial Com Data Input
21	Serial Com Clock
22	Serial Com Select 1
23	Serial Com Select 2

A.3.2.2 JA-2: STEPPER CONNECTOR

The stepper connector is available for future expansion. You must contact Quantum Design before you attempt to use the connector.

A.3.2.3 JA-3: MOTOR SYNC CONNECTOR

This connector outputs the motor encoder position as a high-speed digital serial signal.

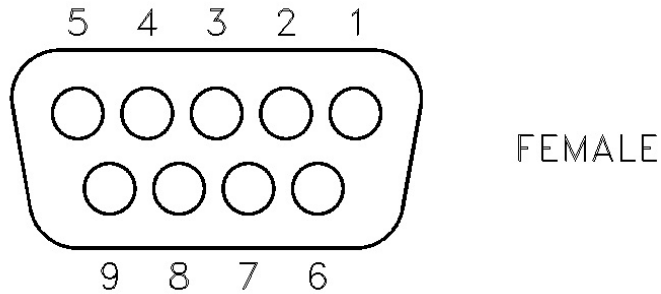


Figure A-4. JA-3: Motor sync connector for the Model CM-A motor module

Table A-4. JA-3: Motor sync connector for the Model CM-A motor module

PIN	FUNCTION
1	Sync +
2	Data +
3	Clock +
4	PSync +
5	Ground
6	Sync –
7	Data –
8	Clock –
9	PSync –

A.3.2.4 JA-4: AUX CONNECTOR

This connector provides three analog inputs and three digital I/O lines for future options.

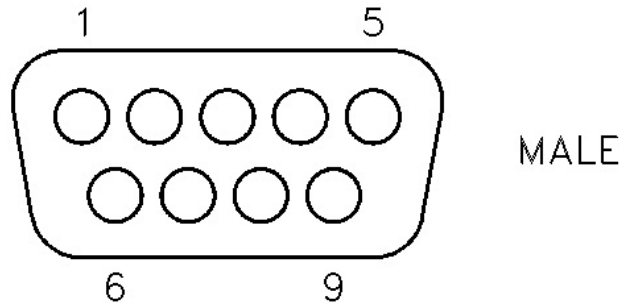


Figure A-5. JA-4: Aux connector for the Model CM-A motor module

Table A-5. JA-4: Aux connector for the Model CM-A motor module

PIN	FUNCTION
1	+5 V
2	Digital I/O P3.10
3	n.c.
4	Analog Input P5.13
5	Ground
6	Digital I/O P3.11
7	Digital I/O P3.8
8	Analog Input P5.12
9	Analog Input P5.14

A.3.2.5 JA-5: MONITOR BNC

This connector is for use by Quantum Design. It can be configured to output signals for diagnostic purposes. By default, the firmware is configured to output the motor position here.

A.4 Model CM-A Sample Motor Module: Rear Panel

The rear panel of the module contains an address selector, a single-guide hole, and the CAN connector through which the module sends and receives network data and receives power.

A.4.1 Address Selector

Each module on the CAN bus must have a unique 5-bit binary address. The selector on the back panel is used to set the four least significant bits, and an internal jumper sets the most significant bit. If the selector is set to zero (0), the module uses its default address. For a Model CM-A motor module, the default address is 10 (or equivalently, “A” on the selector).

A.4.2 Single Guide Hole

The single guide hole is used to align the connector with one of the back-row (high-power) receptacles on the Model 1000 modular control system.

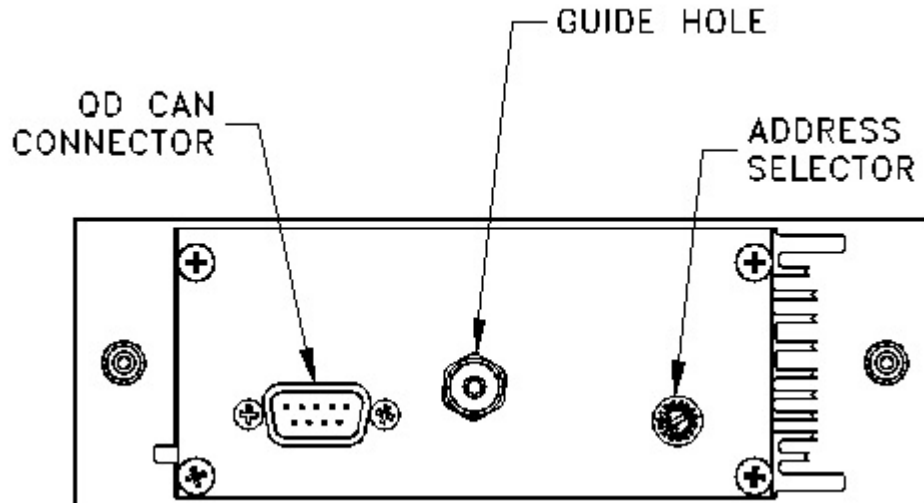


Figure A-6. Rear panel of the Model CM-A motor module

A.4.3 QD CAN Connector

The QD CAN connector is the main communication connection for controlling the module. The CAN network signals (CAN High, CAN Low) are connected to all other CAN modules on the bus and to the PC. Power (± 24 volts), reset, and sync signals are also provided to the module through this connector.

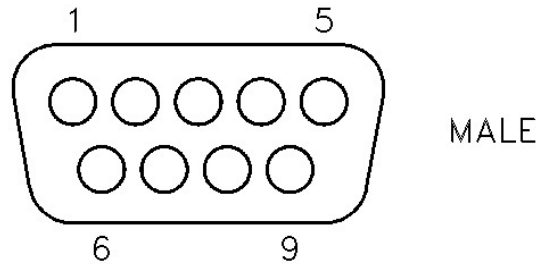


Figure A-7. QD CAN connector on the rear of the Model CM-A motor module

Table A-6. QD CAN connector on the rear of the Model CM-A motor module

PIN	FUNCTION
1	-24 V
2	CAN Low
3	Power Return (24V)
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High / Reset
9	+24 V DC

A.5 Maintenance

(Service Note 1096-307)

The CAN motor module CM-A (QD part number 4101-100), which is used to drive the linear motor of the ACMS II option, dissipates a significant amount of heat when providing the large currents (sometimes over 1 amp) to the motor drive coil. Therefore, it is critical that adequate air cooling is supplied by the fan on the back of the Model 1000 Modular Control Center. If the motor module overheats, the current output is turned off abruptly until the amplifiers cool sufficiently, at which point the current is turned back on. This causes erratic behavior of the motor and presents a hazard to the user. It might also result in damage to the hardware or your research sample.

To help prevent such overheating effects, please follow these guidelines:



WARNING!

As with any CAN module, turn off the power to the Model 1000 before inserting or removing the motor module.

- Regularly—every few months—clean the filter of the air intake fans on the back of the Model 1000. In the case of the upper fan for the module cooling (this is the most critical fan), remove the filter by first pulling off the black plastic guard. The lower fan, for the power supply drawer, can be cleaned by turning off the Model 1000 and vacuuming the outside of the filter.
- Maintain a laboratory temperature below 25 °C.
- Keep the Model 1000 at least 25cm away from walls so that air flow is not impeded.
- Cooling air inside the Model 1000 flows upward past the module and exits at the grate by the front plate of the modules. Make sure these grates are unobstructed.
- Try to minimize the dust level in the lab and keep the floors clean.
- Connect all cables before activating the ACMS II option, and do not unplug the motor drive cable while the option is activated. Reconnecting the cable in this state can lead to motor malfunction.

Quantum Design is continually working to improve the handling of error conditions such as this by enhancing the software, module firmware, and module hardware. Updates to option software (such as the PPMS ACMS II option), new service notes, and application notes are posted on our website www.qdusa.com. Firmware and hardware updates are handled on an individual basis by Quantum Design service.

If you are encountering performance problems with your motor module after observing the above maintenance steps, please contact your local Quantum Design service representative.

Model CM-M ACMS II Module

B.1 Introduction

This appendix contains the following information:

- Section B.2 provides a functional overview of the Model CM-M ACMS II module, including a block diagram and electrical specifications.
- Section B.3 describes the front panel and relevant components of the Model CM-M ACMS II module.
- Section B.4 describes the back panel and relevant components of the Model CM-M ACMS II module.

B.2 Functional Overview

The Model CM-M (4101-432) is a synchronous detection module that performs the real-time signal processing for the ACMS II option. The module and its front panel are shown in Figure B-1.

The principle function of this module is to detect the in-phase and quadrature-phase components of one or two input signals (e.g., pickup coils), as well as a digital reference from, say, a position encoder. The detection is done by multiplying each of the signals by both a sine function and a cosine function. These sine components are computed once per cycle and can be output at this rate, or they can be averaged for multiple cycles with statistics calculated for the ensemble of measurements. Other features include a thermometer bridge circuit for temperature measurements, two programmable gain amplifiers, in-system programmable on-board flash memory for program storage, and a serial ROM for calibration and other configuration data.

The module is designed to plug into the Model 1000 modular control system or an equivalent host chassis that can provide power and the required CAN network signals to communicate with the module.

B.2.2 Specifications

Table B-1. Electrical specifications for the Model CM-M ACMS II detection module

Input Ranges (from preamp)	5 V, 0.5 V, 50 mV, 5 mV
Thermometer Current Ranges	±0.02 mA, ±0.5 mA
Thermometer Voltage Range	±10 mV
Thermometer Sample Rate	14 Hz
Thermometer Resistance Range	20 to 200,000 ohms
Supply Voltage	±24 V DC

B.3 Model CM-M Detection Module: Front Panel

B.3.1 Indicator LEDs

The front panel of the Model CM-M detection module has two LEDs in the top left, as shown in Figure B-1. The PWR LED indicates the power-on status of the module. The COP (CANopen Protocol) LED indicates the status of the CAN network controller. Table B-2 outlines the LED states and provides solutions in the event of a problem.

Important: The error information in Table B-2 refers to situations that persist for longer than about 15 seconds. Typically, when the module is powered on, the LEDs briefly flash red before they turn green. This is a normal part of the startup or reset sequence.

Table B-2. LED guide for the Model CM-M ACMS II detection module

LED	COLOR	STATUS	MEANING AND/OR SOLUTION
PWR	Green	On	The processor is running with no errors (normal)
	Red	Flashing	Errors were encountered during the self-test. The flashing sequence can be used to determine the cause of the failure.
COP	Green	On	CAN status is operational (normal)
		Flashing	CAN status is pre-operational. Verify that cable is connected to PC.
	Red	On or flashing	Error on the CAN bus. Contact Quantum Design for assistance.

If you are unable to achieve operation with both LEDs green, please contact Quantum Design for assistance.

B.3.2 Connectors and Pinout Tables

B.3.2.1 JB-1: MOTOR SYNC CONNECTOR

This connector reads the motor encoder position from the Model CM-A VSM motor module as a high-speed digital serial signal.

Important: This sync connector and the short crossover sync cable (3096-400) to module CM-A are no longer used in newer versions of firmware in modules CM-A and CM-B (after ca. 2009).

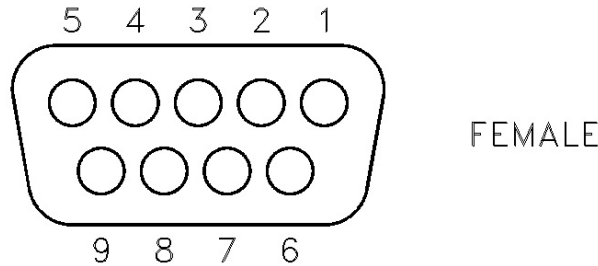


Figure B-3. JB-1: Motor sync connector for the Model CM-B VSM detection module

Table B-3. JB-1: Motor sync connector for the Model CM-B VSM detection module

PIN	FUNCTION
1	Sync +
2	Data +
3	Clock +
4	PSync +
5	Ground
6	Sync -
7	Data -
8	Clock -
9	PSync -

B.3.2.2 JB-2: MONITOR BNC

This connector outputs the amplified pickup coil signal.

B.3.2.3 JB-3: PREAMP CONNECTOR

This connector is the main connection to the preamplifiers and coilset puck. It contains two analog inputs for the synchronous detection, current and voltage for a thermometer, power for the preamp, and serial communications to the serial ROM in the preamp box for storing calibration or configuration information.

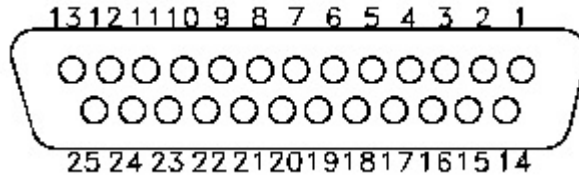


Figure B-4. JB-3: Preamp connector for the Model CM-B VSM detection module

Table B-4. JB-3: Preamp connector for the Model CM-B VSM detection module

PIN	FUNCTION
3	Serial Com Select
4	Serial Com Data MOSI
5	Thermometer Current +
6	Thermometer Voltage +
9	+15 V
10	Channel 1 Input +
11	+5 V
12	Channel 2 Input +
13	Signal Ground
16	Serial Com Clock
17	Serial Com Data MISO
18	Thermometer Current -
19	Thermometer Voltage -
22	-15 V
23	Channel 1 Input -
25	Channel 2 Input -

B.4 Model CM-M Detection Module: Rear Panel

The rear panel of the Model CM-M VSM detection module contains an address selector, two guide holes, and the CAN connector through which the module sends and receives network data and receives power.

B.4.1 Address Selector

Each module on the CAN bus must have a unique 5-bit binary address. The selector on the back panel is used to set the four least significant bits, while an internal jumper sets the most significant bit. If the selector is set to “0,” the module uses its default address. For a Model CM-B VSM detection module, the default address is 8.

B.4.2 Guide Holes

The two guide holes are used to align the connector with either a low-power receptacle or a high-power receptacle on the Model 1000 modular control system.

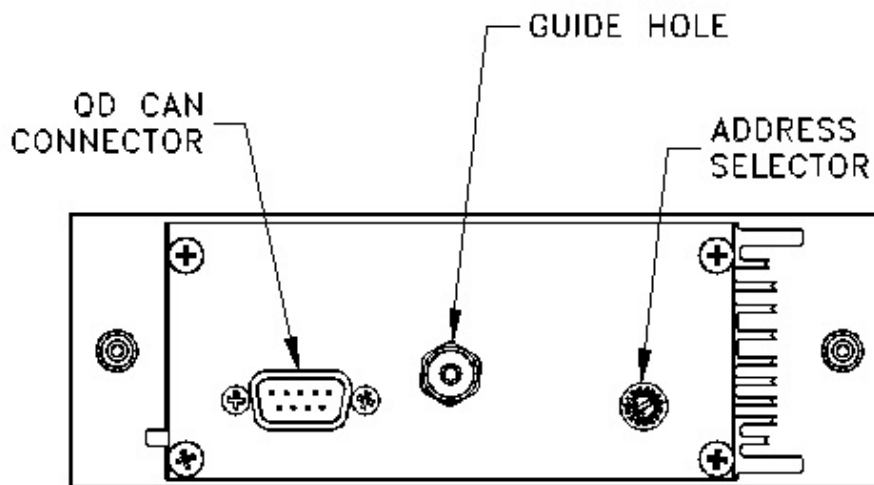


Figure B-5. Rear panel of the Model CM-M ACMS II detection module

B.4.3 QD CAN Connector

The QD CAN connector is the main communication connection for controlling the Model CM-B VSM detection module. The CAN network signals (CAN High, CAN Low) are connected to all other CAN modules on the bus and to the PC. Power (± 24 volts), reset, and sync signals also are sent to the module through this connector.

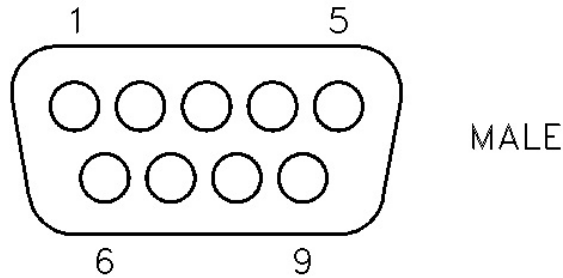


Figure B-6. QD CAN connector on rear of the Model CM-M ACMS II module

Table B-5. QD CAN connector on the rear of the Model CM-M ACMS II module

PIN	FUNCTION
1	-24 V
2	CAN Low
3	Power Return (24V)
4	Sync Low
5	Line Sync
6	System Ground
7	CAN High
8	Sync High / Reset
9	+24 V DC

