Quantum Design

Vibrating Sample Magnetometer (VSM) Option User's Manual

Part Number 1096-100, B0

Trademarks

All product and company names appearing in this manual are trademarks or registered trademarks of their respective holders.

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

$$C\hspace{-0.3em}\in$$

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.

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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 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.

o Chapter 7 describes some troubleshooting strategies for VSM measurements.

o Appendix A provides a functional description of the Model CM-A VSM motor module, including diagrams and electrical specifications.

o Appendix B provides a functional description of the Model CM-B VSM detection module, including diagrams and electrical specifications.

o 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.
`.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.

C A U T I O N !

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

W A R N I N G !

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

W A R N I N G !

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

C H A P T E R   1

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:

$$V_{coil} = \frac{d\Phi}{dt}$$
$$= \left(\frac{d\Phi}{dz}\right)\left(\frac{dz}{dt}\right)$$

(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:

$$V_{coil} = 2\pi f C m A \sin(2\pi f t)$$

(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.
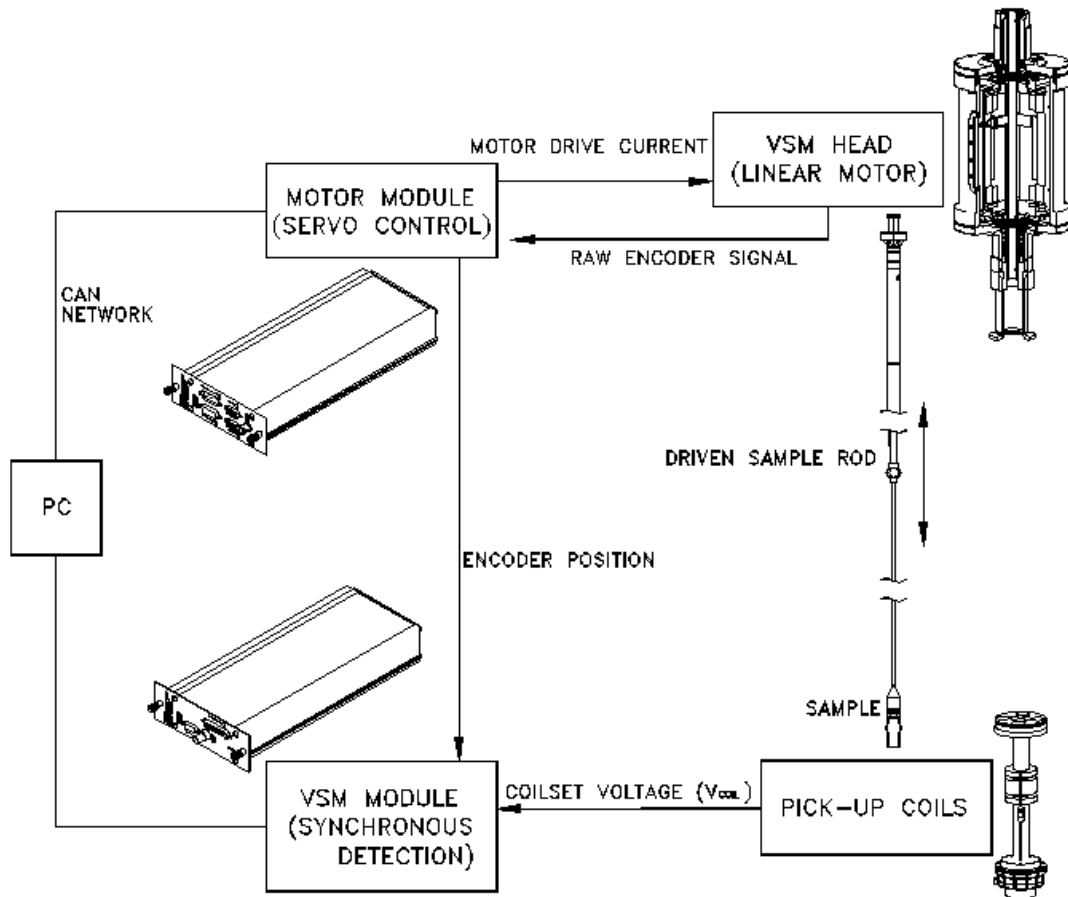
Figure 1-1. Operating principle for the VSM option

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

> **W A R N I N G !**
>
> 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.

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

> **W A R N I N G !**
>
> 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.

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

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[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   Cryogens (if applicable)

<table>
<tr><td>⚠</td><td>

W A R N I N G !

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.

</td></tr>
</table>

❖ Always wear protective clothing, including thermal gloves, eye protection, and covered shoes, when you work with liquid helium, liquid nitrogen, or other cryogens. Avoid loose clothing or loose fitting gloves that could collect cryogenic liquids next to the skin. The extreme cold of liquid and gaseous cryogens 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
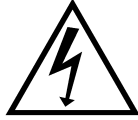
> **W A R N I N G !**
>
> The VSM and the PPMS family of instruments are powered by nominal voltages that range from 100 V to 240 V AC. These voltages are potentially lethal, so you should exercise appropriate care before opening any of the electronics units, including turning off the equipment and disconnecting 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.

C H A P T E R 2

Installing and Removing the VSM Option

2.1 Introduction

This chapter contains the following information:

- Section 2.2 lists the components of theVSM 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.
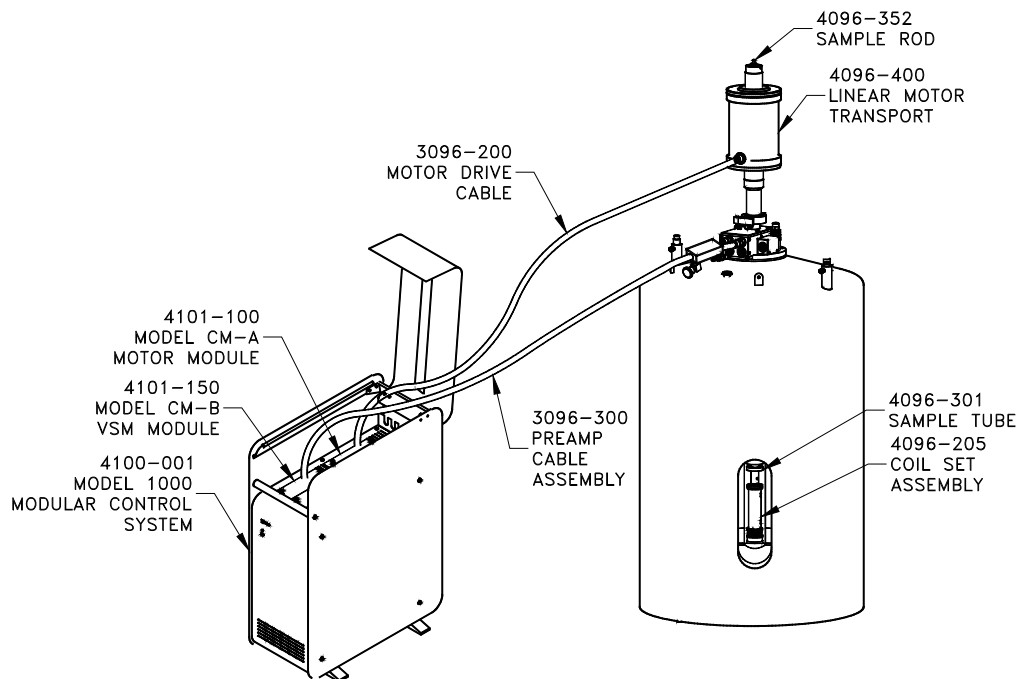
Figure 2-1. System components for PPMS VSM option

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.

> ⚠️ **W A R N I N G !**
>
> 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.

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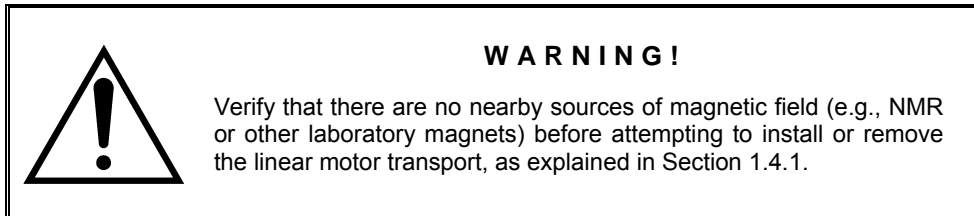
3. Vent the sample chamber.

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 tool[1] 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.

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[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.
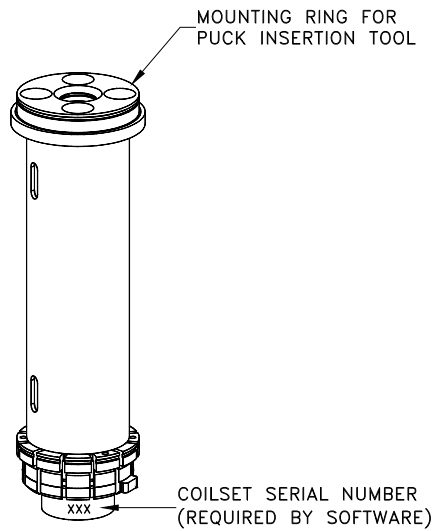
Figure 2-2. Coilset puck (4096-204 or 4096-600), with arrows to coilset serial number and to mounting ring for attaching the puck-insertion tool
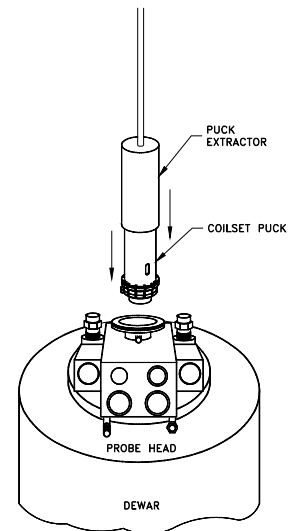
Figure 2-3. Installing the VSM coilset puck with the puck-insertion tool.

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.
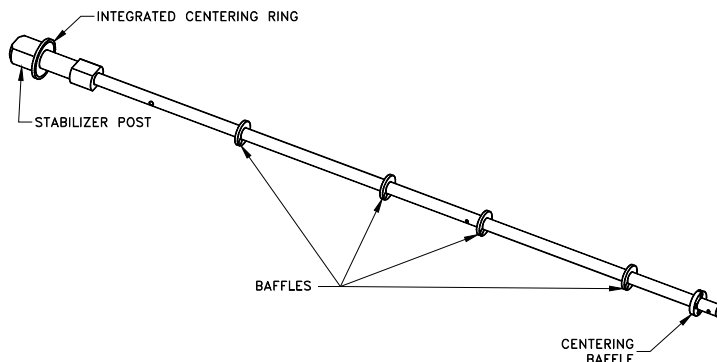
Figure 2-4. VSM sample-tube assembly (4096-301 is shown for example)
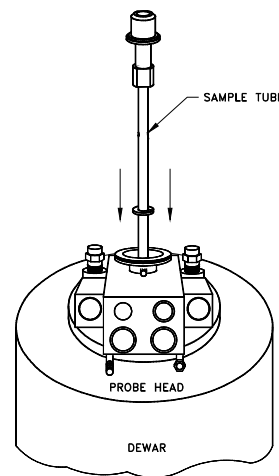
Figure 2-5. Inserting the VSM sample tube into the sample chamber

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

> ⚠ **W A R N I N G !**
>
> 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.
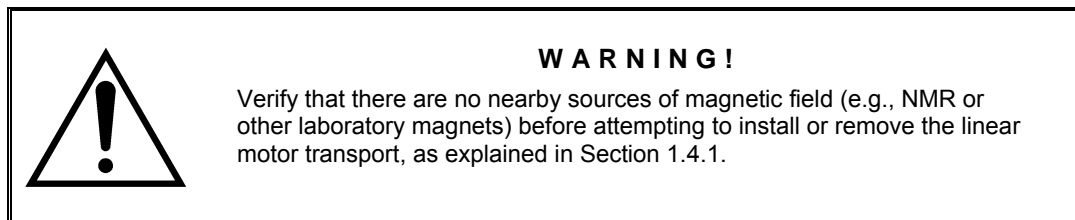
The VSM linear motor transport (Figure 2-6) moves the sample. You will mount the linear motor transport directly on top of the sample-chamber opening after you have inserted the sample tube into the sample chamber. Before you install the linear motor transport, you must remove the shipping plug and install the extender-tube-flange assembly. See Chapter 5 for more information on the linear motor transport and its operations.
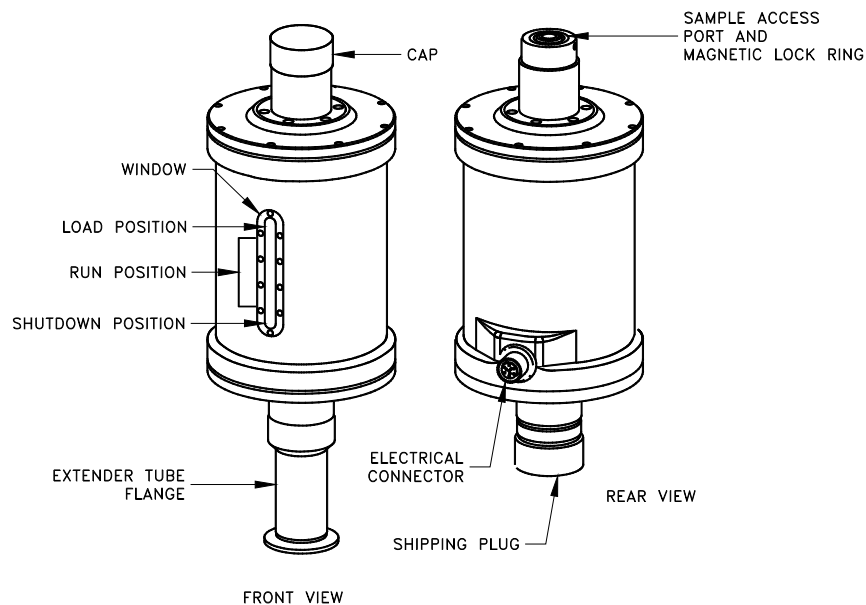
Figure 2-6. Front and rear views of the VSM linear motor transport (4096-400). The rear view (right) shows the transport with the shipping plug installed; in the front view (left), the shipping plug has been replaced by the extender tube flange.

Chapter 2
Section 2.2
Installing and Removing the VSM Option
1BInitial Installation of the Hardware and Software

C A U T I O N !

♦ 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.**
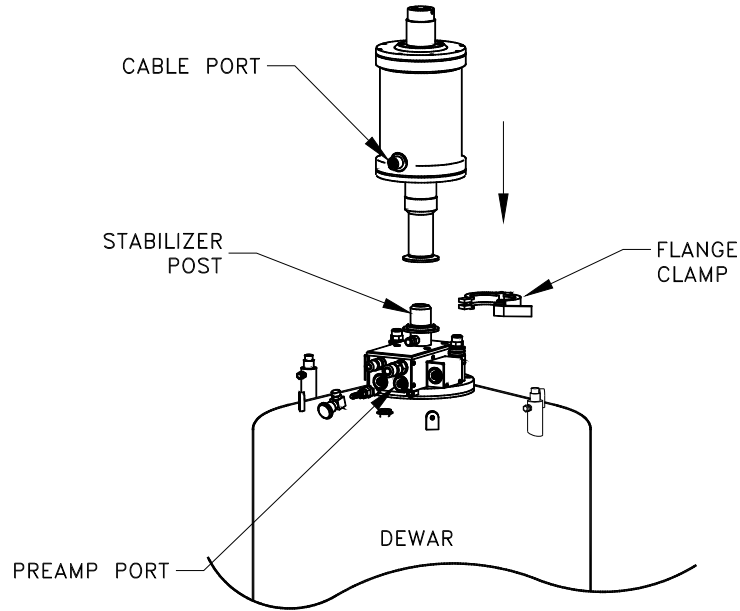
Figure 2-7. Installing the VSM linear motor transport on the top flange

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.
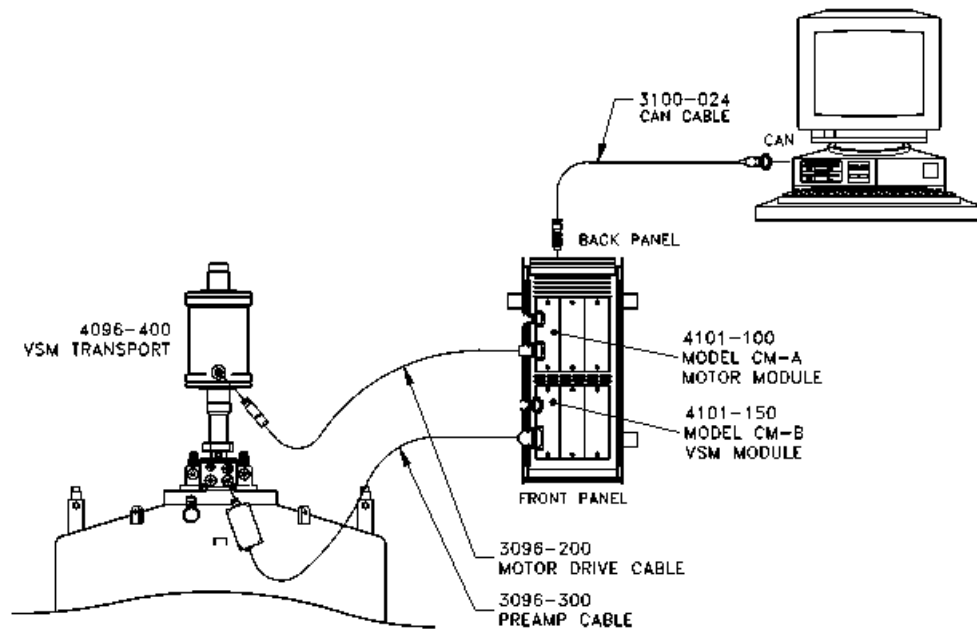
Figure 2-8. VSM option connections on a PPMS

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).
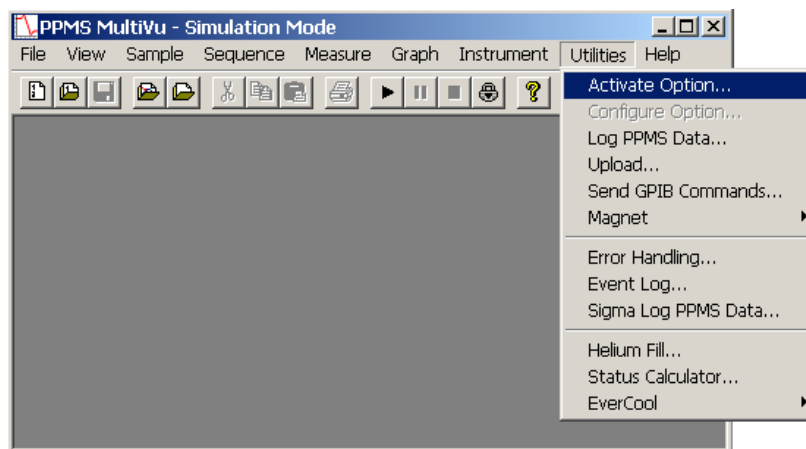
Figure 2-9. PPMS MultiVu menu bar and Utilities dropdown menu with Activate Option selected

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.
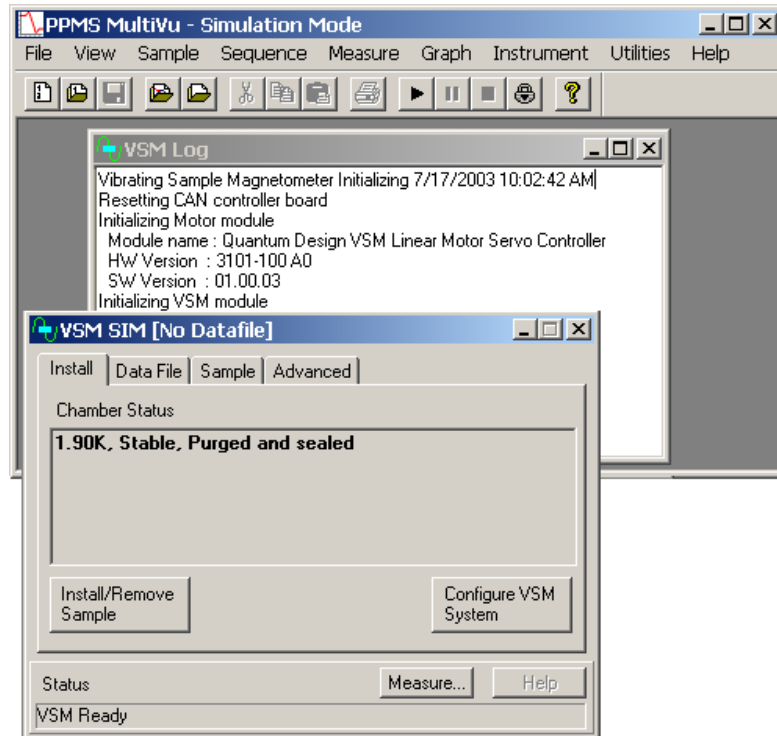
Figure 2-10. VSM Control Center and VSM Log window
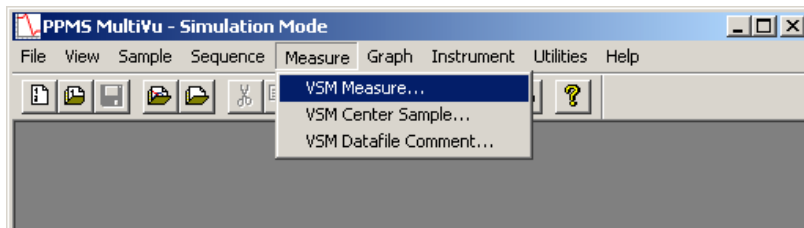
Figure 2-11. PPMS MultiVu menu bar and Measure dropdown menu
for VSM option

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.
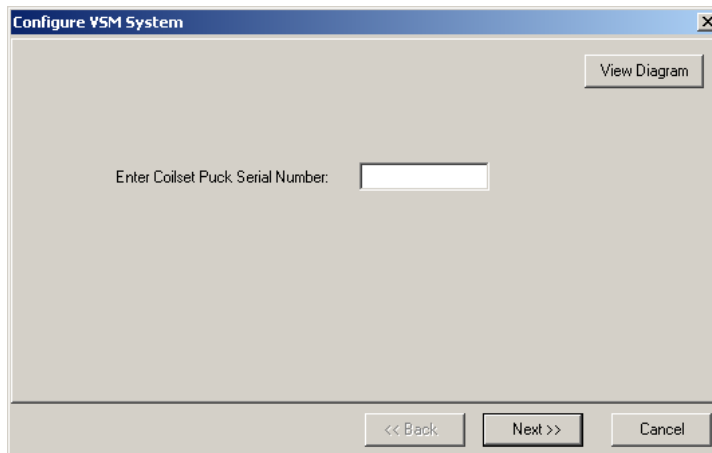
Figure 2-12. Configure VSM System dialog, page 1

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.
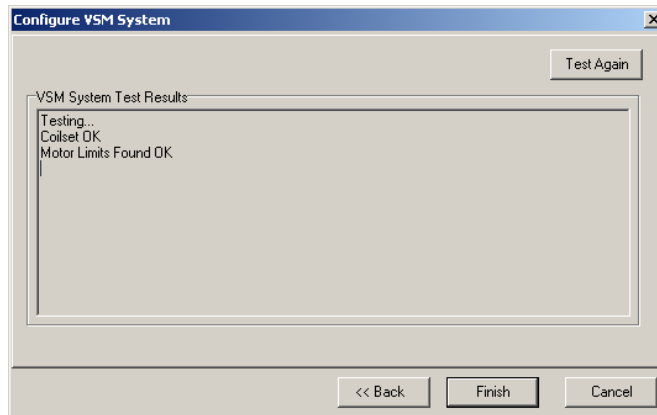
Figure 2-13. Configure VSM System dialog, page 2: Testing calibration file

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

> ⚠ **W A R N I N G !**
>
> 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.

> **C A U T I O N !**
>
> 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).

W A R N I N G !

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.

C H A P T E R   3

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=3.8mm, D=2.8mm) 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 *L* (mm)	Diameter *D* (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	2.0 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
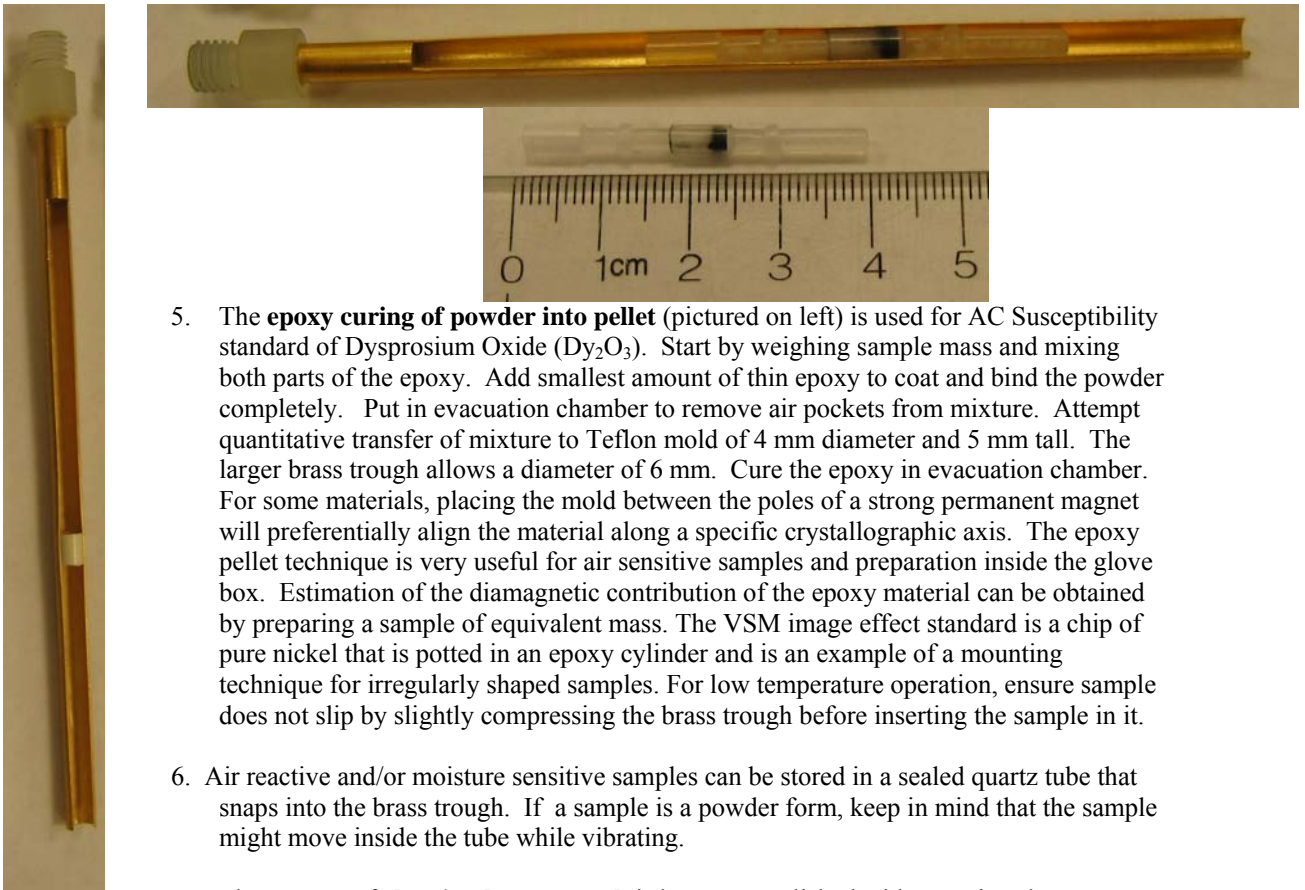
5. The **epoxy curing of powder into pellet** (pictured on left) is used for AC Susceptibility standard of Dysprosium Oxide (Dy_2O_3). Start by weighing sample mass and mixing both parts of the epoxy. Add smallest amount of thin epoxy to coat and bind the powder completely.   Put in evacuation chamber 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.
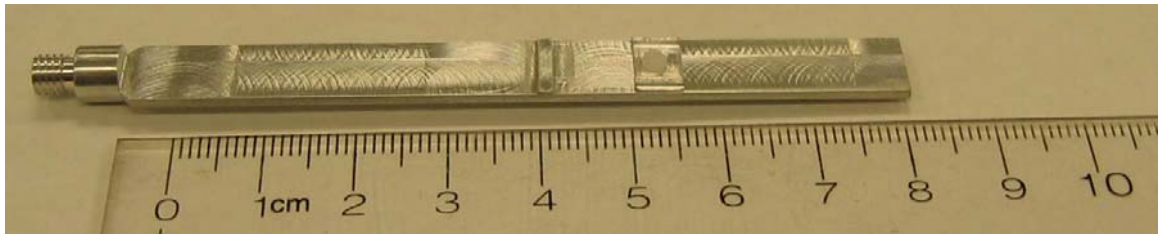
E. **Straws and gelatin capsules are no longer being used**. The commercially available straws are not straight enough to be used for the VSM measurement. The straws tend to bend and make contact with the chamber walls. The frictional heating will restrict the base operational temperature. In addition, 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.

C H A P T E R   4

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:

Measure location = (touchdown location) + (coilset height) – (sample offset),

where coilset height ("H" in Figure 5-4) is a calibrated constant for a given puck and is near 40mm.
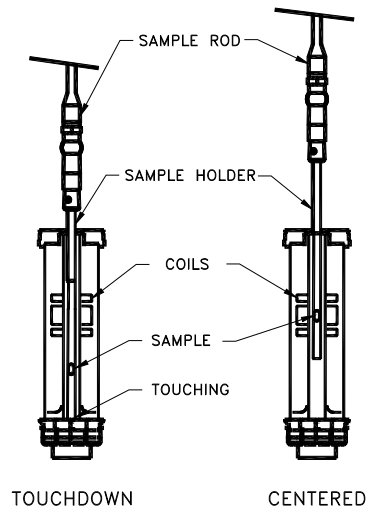
Figure 4-1. The touchdown centering operation

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)

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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).
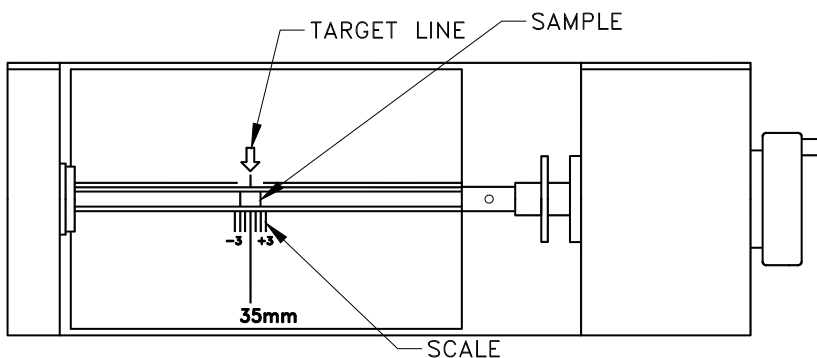
Figure 4-2. Reading the position of the sample from the sample-mounting station

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.
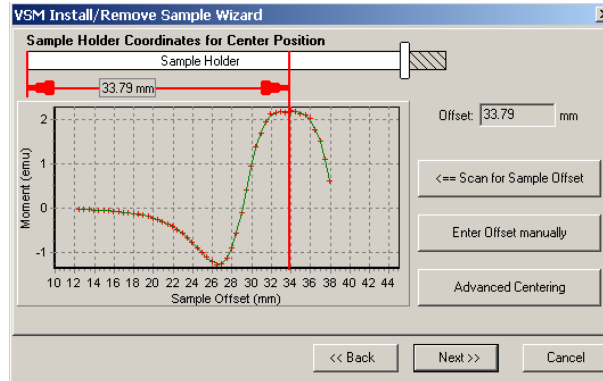
Figure 4-3. Example of a centering scan for a sample with a large
positive magnetic moment of ~2 emu.

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.
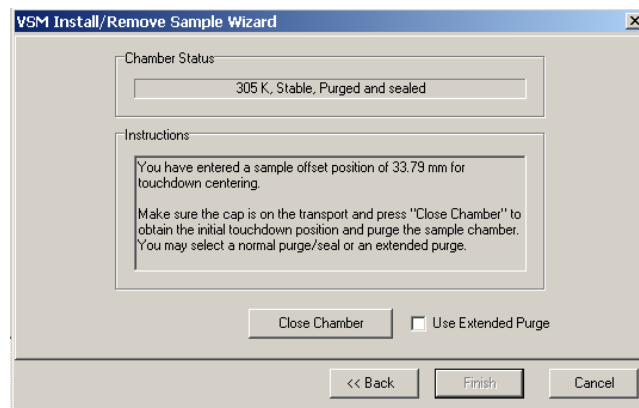
Figure 4-4. The VSM Install wizard, final page

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 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.
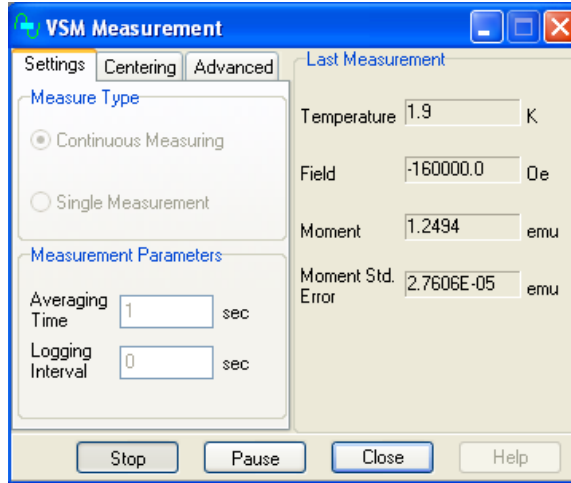
Figure 4-5. VSM Measurement dialog

C H A P T E R   5

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.

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♦ 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.
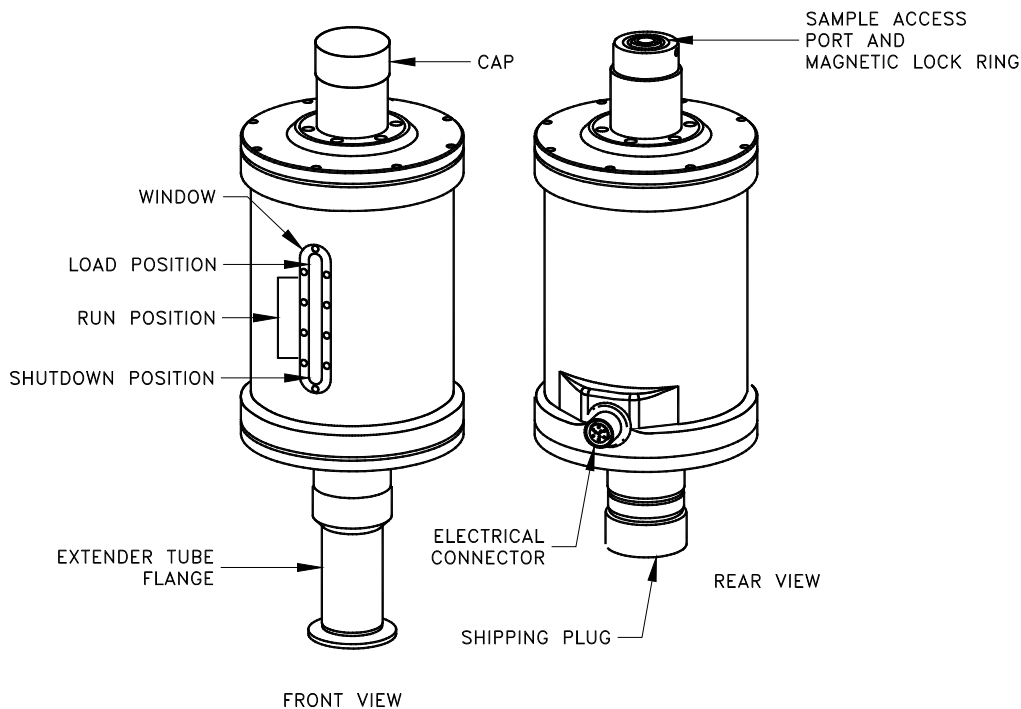
Figure 5-1. VSM linear motor transport (4096-400)

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.

C A U T I O N !

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.
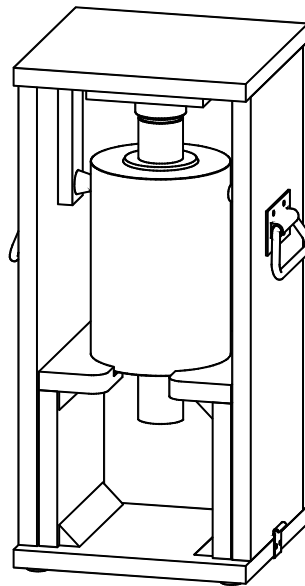
Figure 5-2. Storage case (4096-150) for the linear motor transport.

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*.
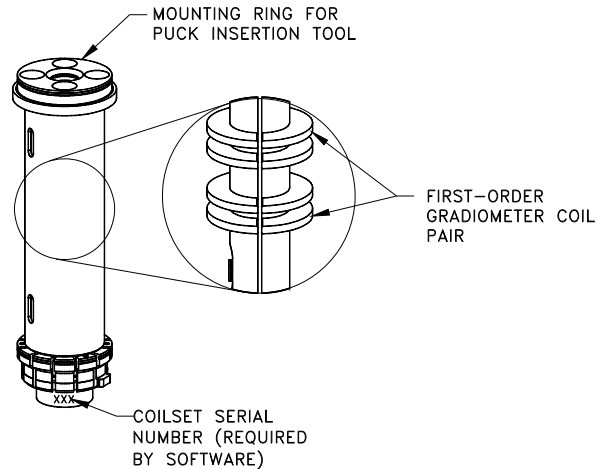
Figure 5-3. Coilset puck (4096-204)
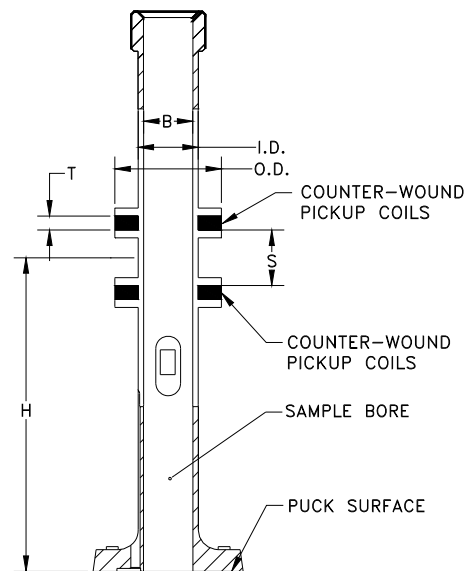
Figure 5-4. The internal dimensions of a standard coilset puck are as follows: Bore diameter (B) = 6.33 mm; coil thickness (T) = 1.78 mm; coil spacing (S) = 7.11 mm; coil inner diameter (ID) = 7.73 mm; coil outer diameter (OD) = 13.7 mm; height above puck surface (H) = 40.1 mm.

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   probe head   JB-3: Preamp connector on Model CM-B VSM detection module
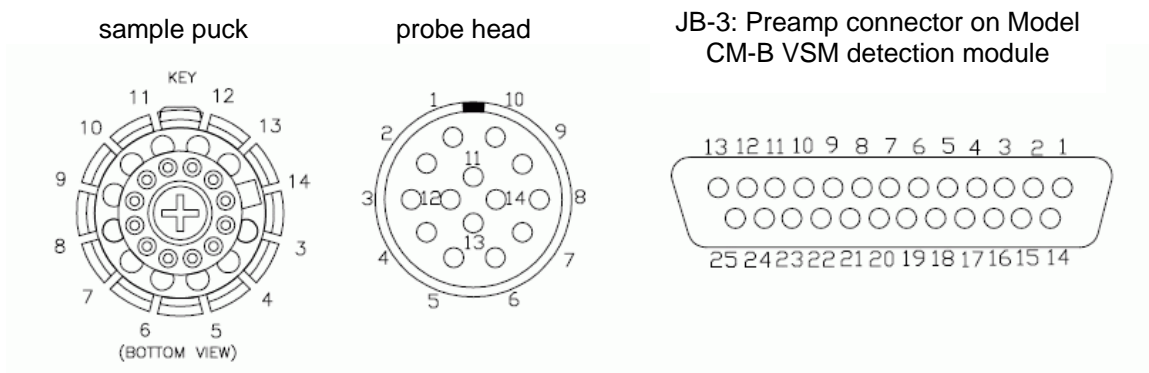
SAMPLE PUCK	GRAY LEMO CONNECTOR AT PROBE HEAD	JB-3 PREAMP ON CM-B VSM MODULE	FUNCTION
3	3	5	Thermometer Current +
4	4	18	Thermometer Current –
5	5	6	Thermometer Voltage +
6	6	19	Thermometer Voltage –
11	11	10	Channel 1 Input +
12	12	23	Channel 1 Input –
13	13	12	Channel 2 Input +
14	14	25	Channel 2 Input –

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.

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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.
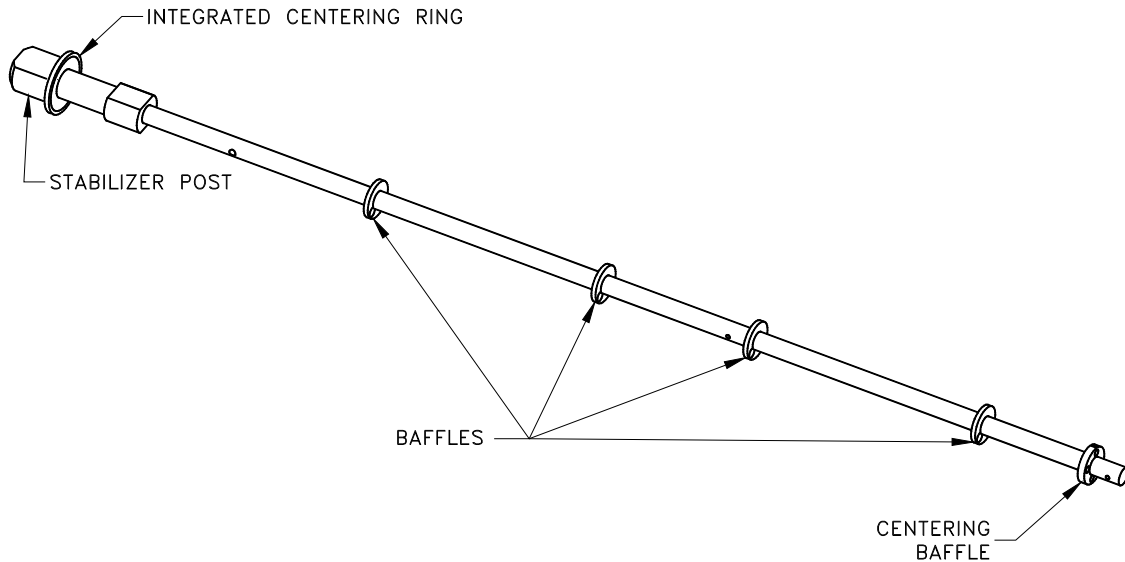
Figure 5-5. VSM sample tube (4096-301 shown for example)

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.
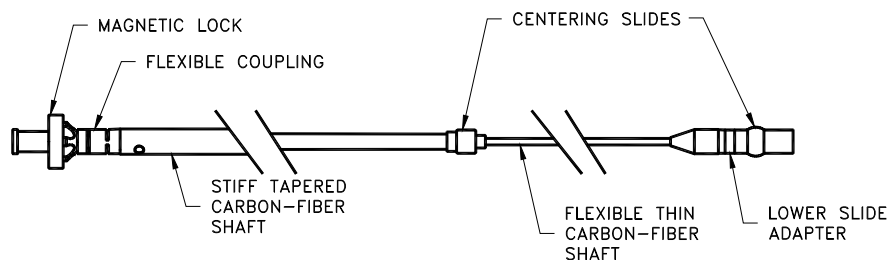
Figure 5-6. VSM sample rod (4096-352 shown for example)

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.
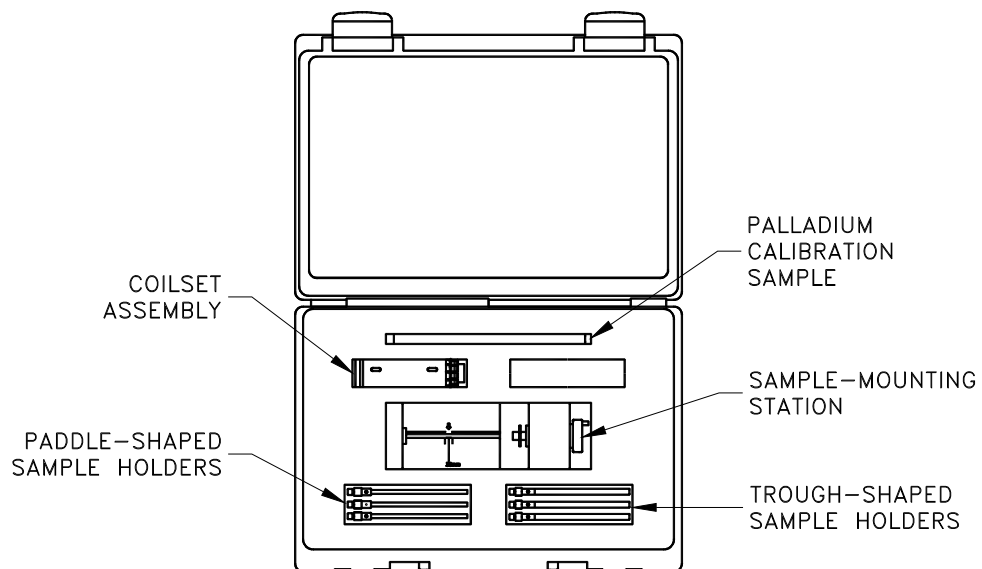
Figure 5-7. VSM Option User's Kit (4096-100)

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).
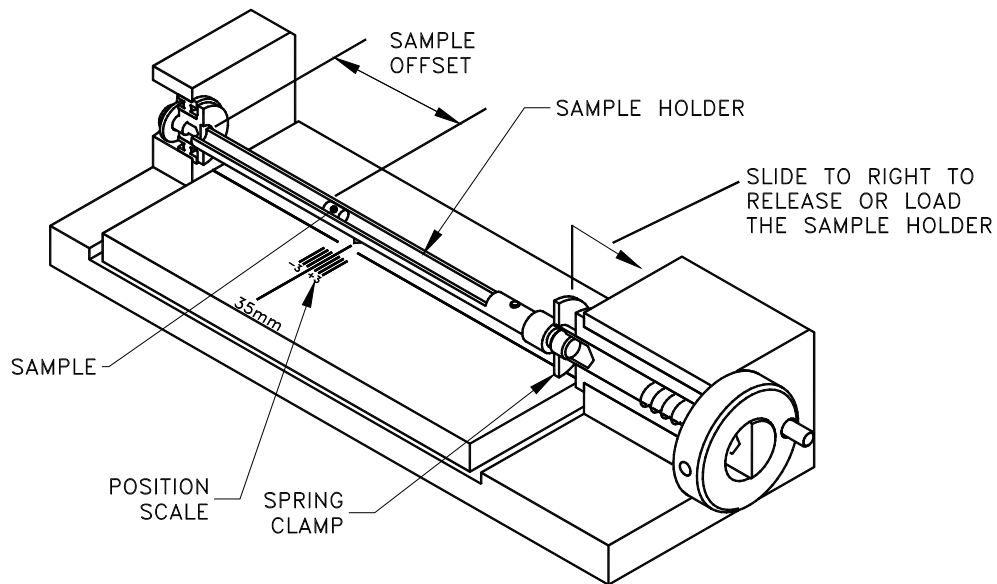
Figure 5-8. Sample-mounting station (4096-110)

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.
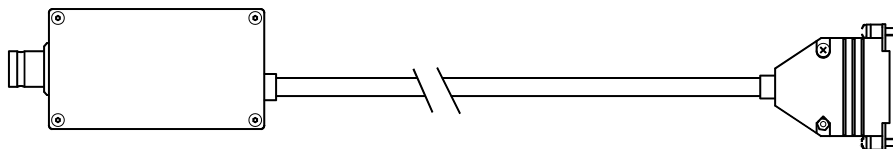
Figure 5-9. Preamp cable assembly (3096-300)

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.
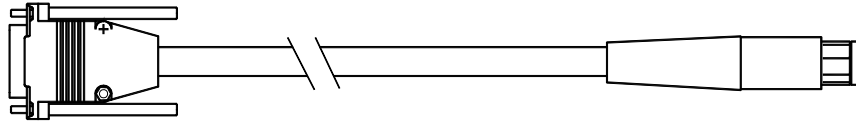
Figure 5-10. Motor drive cable (3096-200)

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.
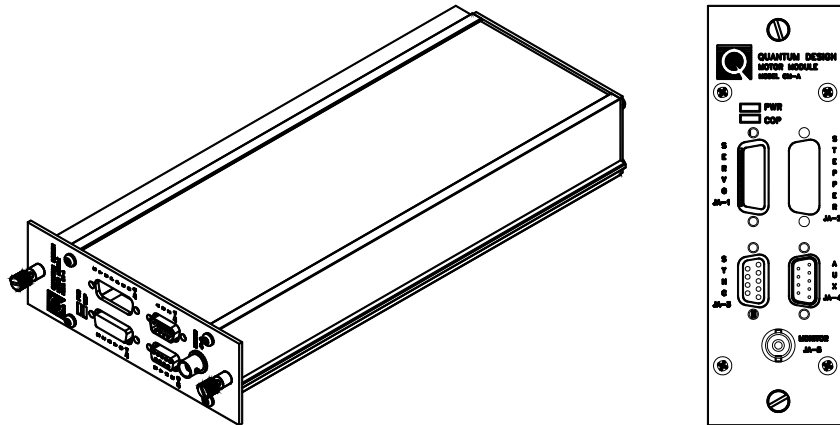
Figure 5-11. Model CM-A VSM motor module (4101-100)

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.
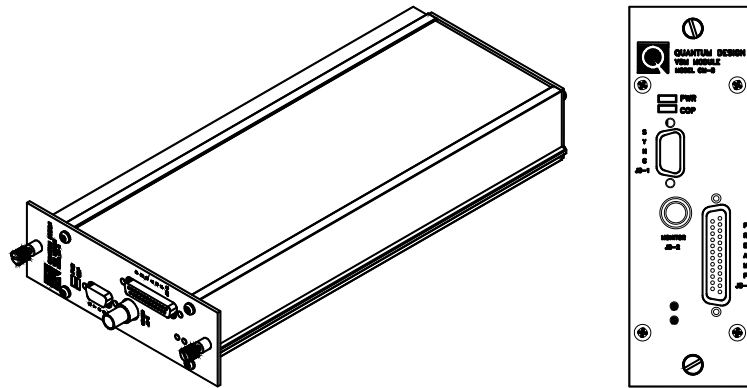
Figure 5-12. Model CM-B VSM detection module (4101-150)

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.
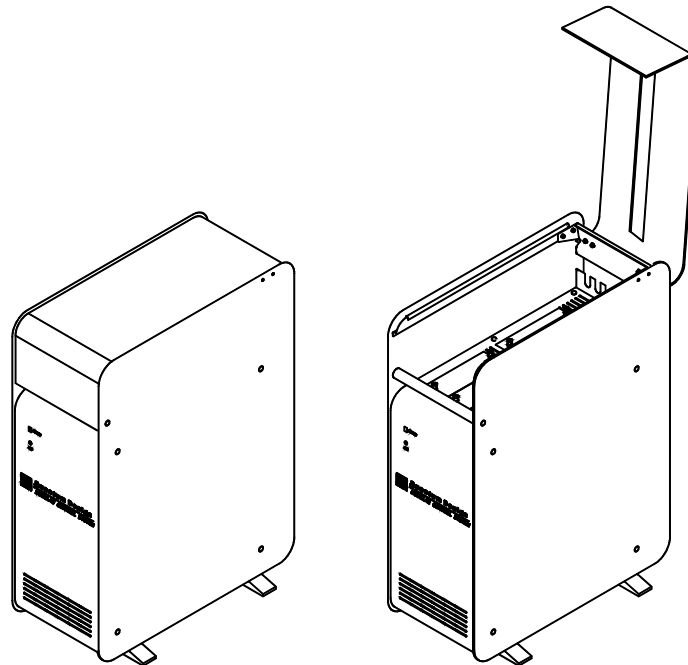
Figure 5-13. Model 1000 modular control system (4100-001)

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.

C H A P T E R   6

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).
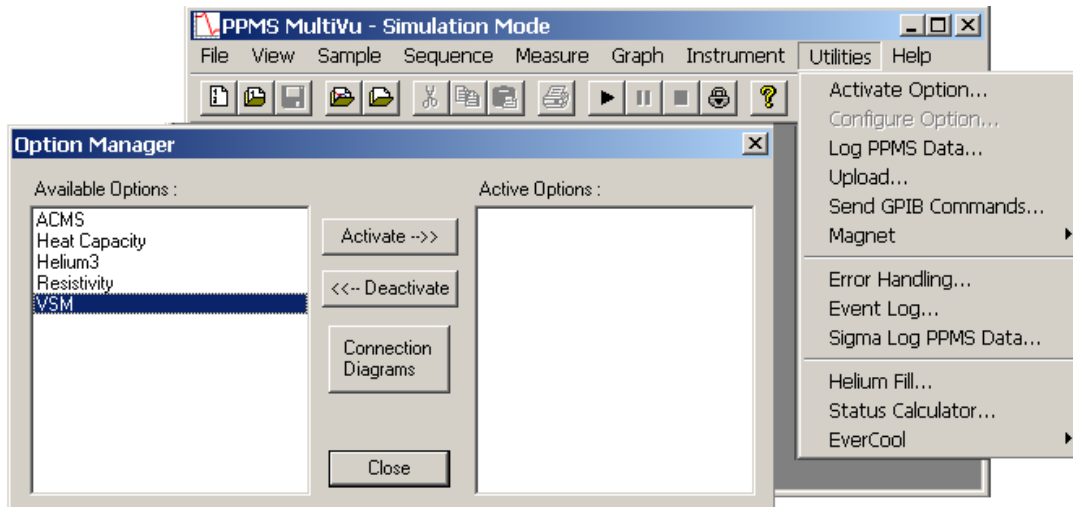
Figure 6-1. Using the MultiVu dropdown Utilities menu and Option Manager to
activate the VSM Option

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.
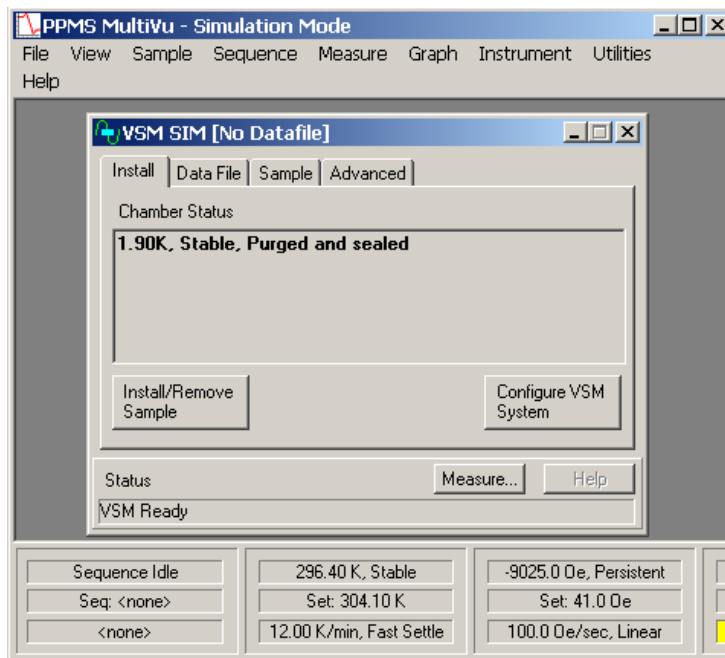
Figure 6-2. The MultiVu window and the VSM Control Center (VSM SIM),
including the Install tab, the VSM Status area, and the MultiVu Status bar. Note
that the VSM Log window has been minimized.

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.
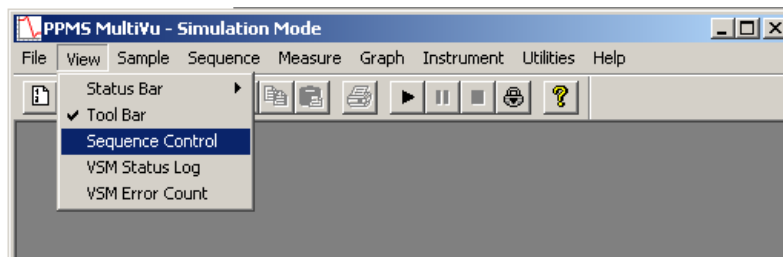
Figure 6-3. The MultiVu window and the View dropdown menu showing VSM immediate-mode commands

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.
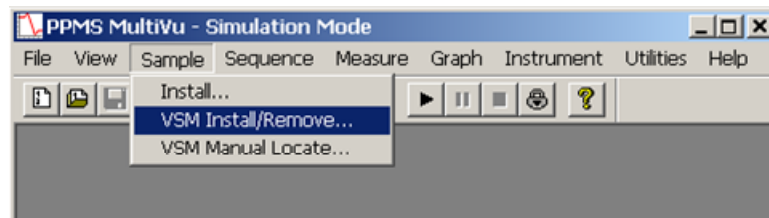
Figure 6-4. The MultiVu window and the Sample dropdown menu showing VSM immediate-mode commands

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.

o 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).
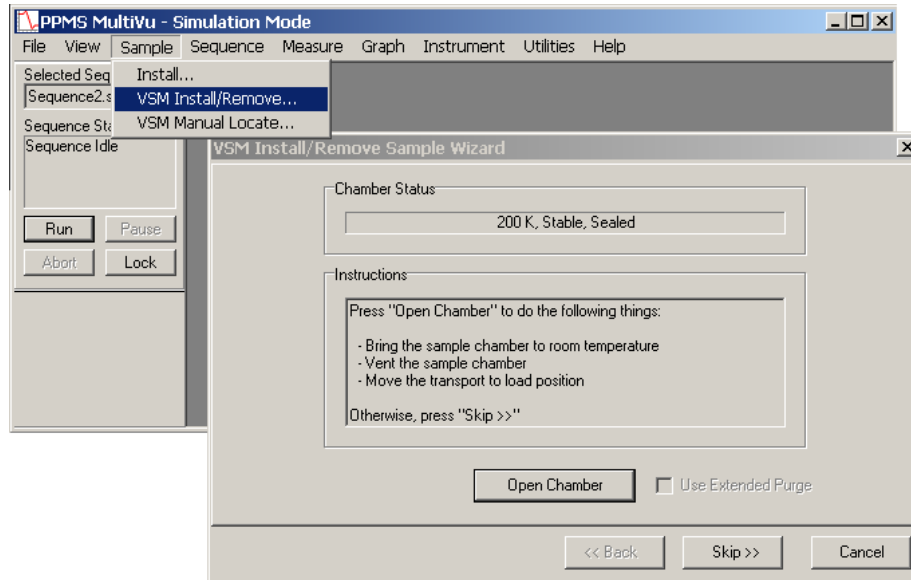
Figure 6-5. The Specify Sample Location dialog that opens from the
dropdown menu when you select Sample >> VSM Manual Locate

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.
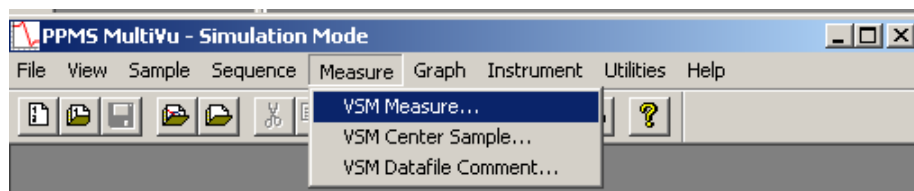
Figure 6-6. VSM immediate-mode commands in the context of the MultiVu window and the
Measure dropdown menu

○ 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).
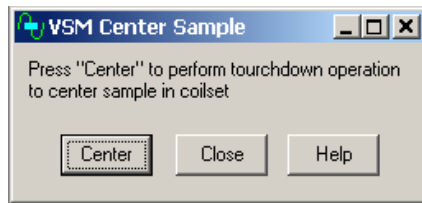
Figure 6-7. The immediate-mode VSM Center Sample popup
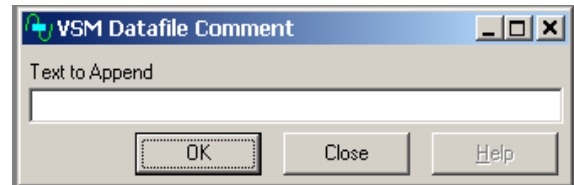
Figure 6-8. The immediate-mode VSM Datafile Comment popup

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
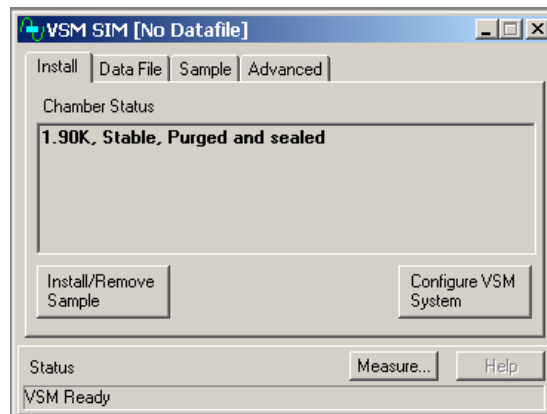
Figure 6-9. Install tab in the VSM Control Center

The **Install** tab provides access to two wizards.

- o **Install/Remove Sample** guides the user through installation of a sample into the VSM measurement space and is outlined in Section 4.3.3.

- o **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 .
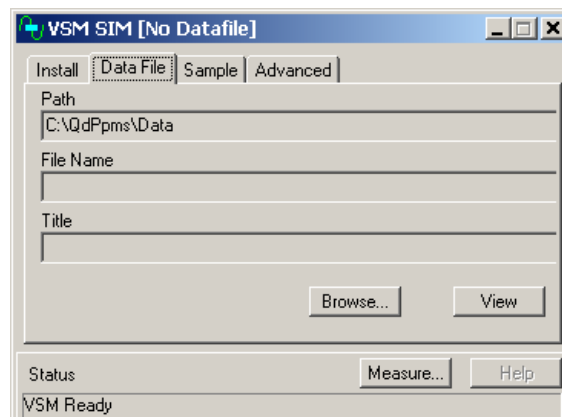
Figure 6-10. Data File tab in the VSM Control Center

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.
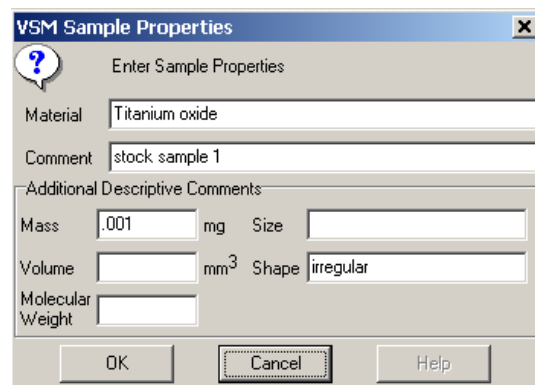
Figure 6-11. Recording sample properties

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.
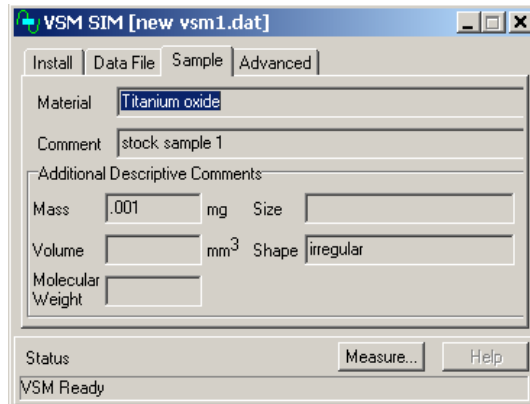
Figure 6-12. Sample tab in the VSM Control Center

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-m^2 (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.
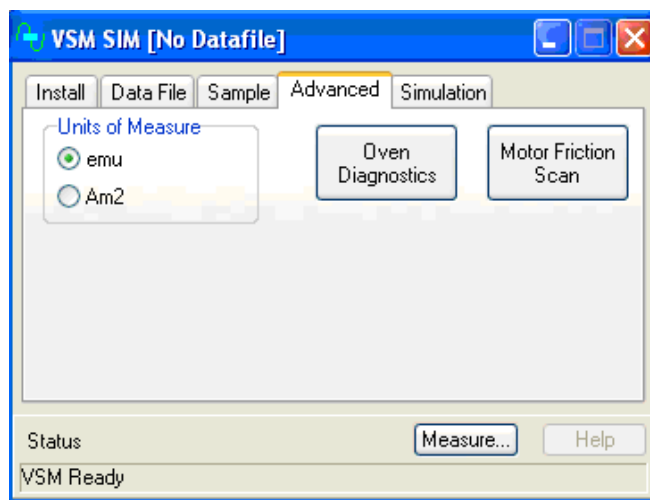
Figure 6-13. Advanced tab in the VSM Control Center

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** 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.
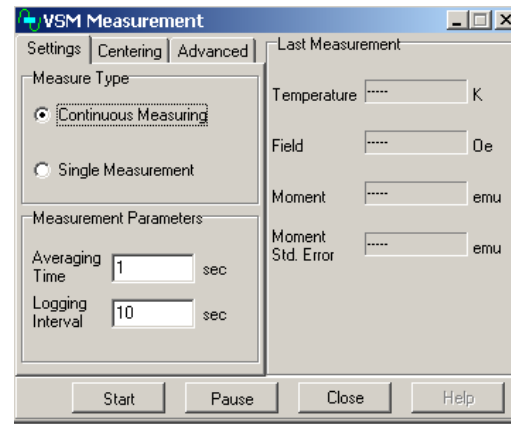
Figure 6-14. VSM Measurement dialog and the Settings tab

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.
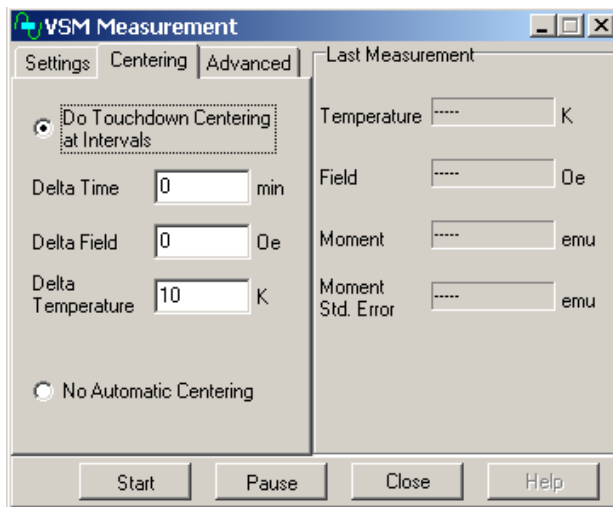
Figure 6-15. Centering tab in the VSM Measurement dialog

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**.
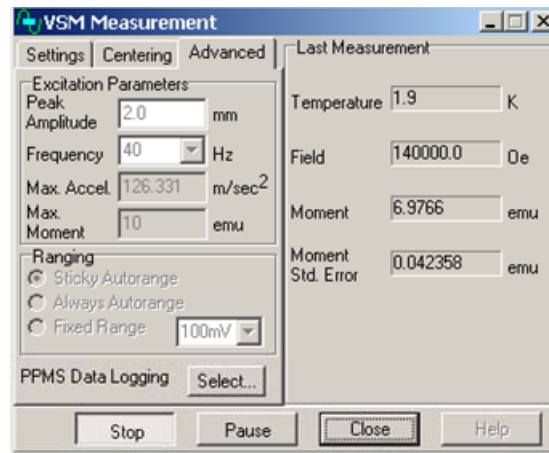
Figure 6-16. Advanced tab in the VSM Measurement dialog

Excitation Parameters

The excitation parameters are explained by the equation:

Position(time) = [Peak Amplitude] × sin(2π*[Frequency]×time)

o   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.

o   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.

o   **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.

o   **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.

o   **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.

o   **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.

o   **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.
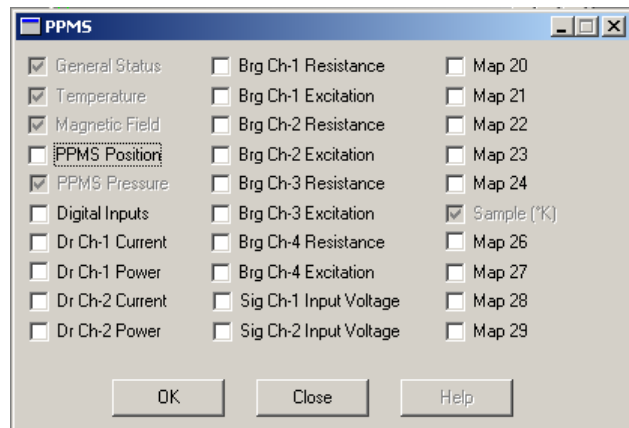
Figure 6-17. Data-logging dialog for selecting additional types of data to be collected

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.

Section 6.8
Sequence-Mode VSM "Moment vs. Field"Command

Chapter 6
VSM Software

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.
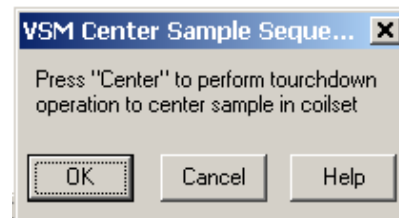
Figure 6-18. Sequence-mode VSM Center Sample command: VSM Center Sample Sequence popup

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.
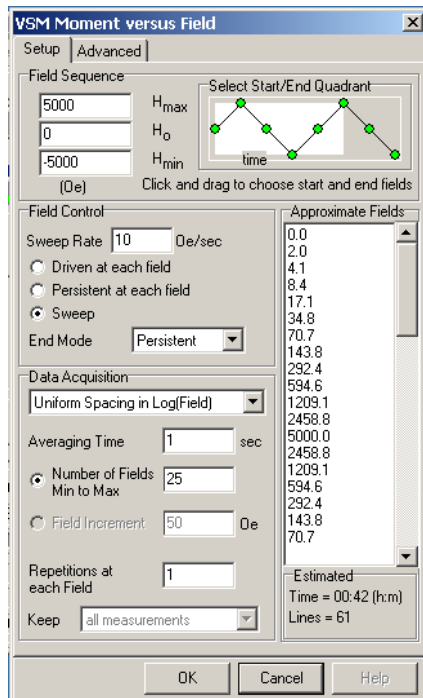
Figure 6-19. Setup tab of the sequence-mode VSM Moment versus Field dialog
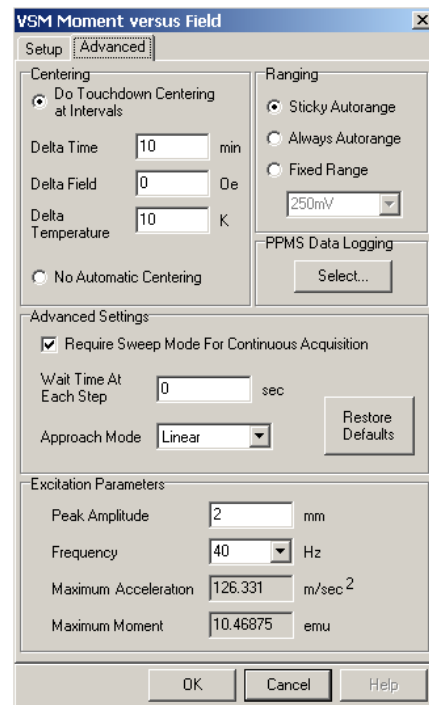
Figure 6-20. Advanced tab of the sequence-mode VSM Moment versus Field dialog

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.

Section 6.8
Sequence-Mode VSM "Moment vs. Field" Command

Chapter 6
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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.
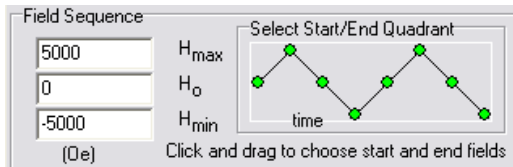
Figure 6-21. Field Sequence section of the VSM Moment vs Field Setup tab: Start-End Quadrant de-selected
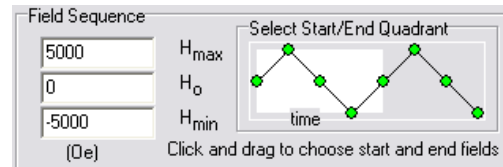
Figure 6-22. Field Sequence section of the VSM Moment vs Field Setup tab: Start-End Quadrant selected

- ○ **Hmax** defines the maximum field used in the measurement.

- ○ **Ho** defines a starting or ending field used in the measurement. By default, **Ho** is the starting point of the measurement, but you also can use **Hmax** or **Hmin** as the starting point.

- ○ **Hmin** defines the minimum field used in the measurement.

   Note: Uniform spacing is referenced between **Hmax** and **Hmin**. This can affect whether **Ho** 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.
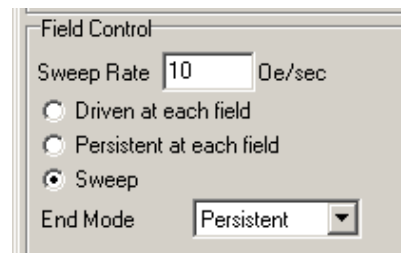
Figure 6-23. Field Control section of the VSM Moment versus Field dialog

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**.

C A U T I O N !

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.

Figure 6-24. Data Acquisition: Data spacing selection

○   **Uniform Spacing in Field^2** 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.

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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.

Figure 6-25. Data Acquisition: Number of Fields and Field Increment

○ 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$_o$** might not be used in the measurement if the combination of uniform spacing and number of fields does not line up evenly with **H$_o$**. If this occurs, **H$_o$** 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.
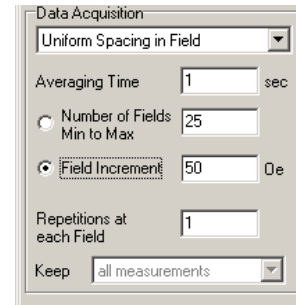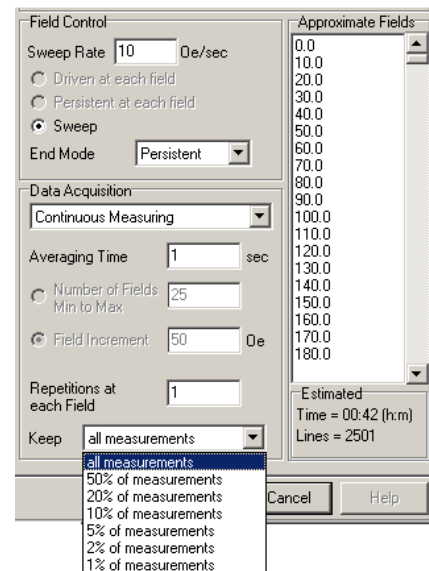
Figure 6-26. Data Acquisition: Keep and Approximate Fields settings

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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.
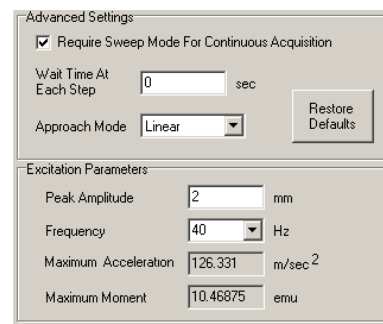
Figure 6-27. Advanced Settings–Advanced tab: Require sweep, Wait Time, and Approach Mode settings

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Sequence-Mode VSM "Moment vs. Temp"Command

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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.
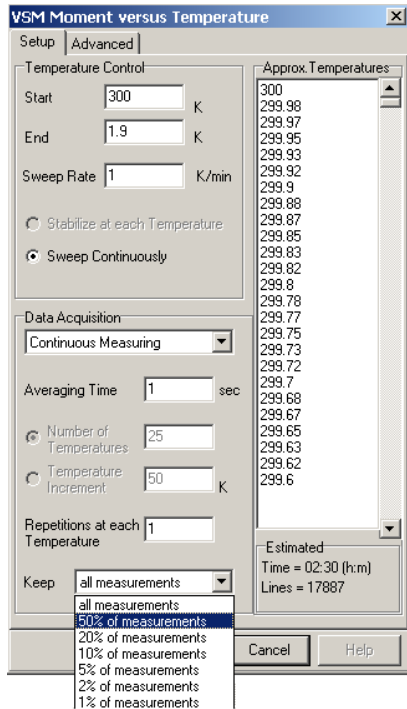
Figure 6-28. Setup tab of the sequence-mode VSM
Moment versus Temperature dialog
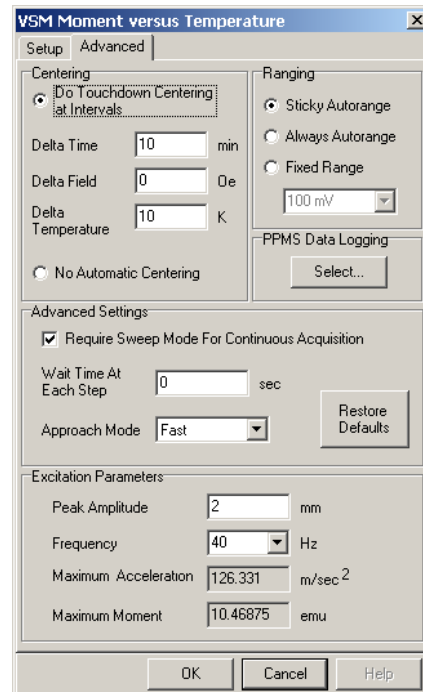
Figure 6-29. Advanced tab of the sequence-mode
VSM Moment versus Temperature dialog

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.
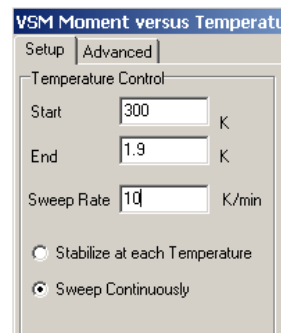
Figure 6-30. Temperature Control section of
the sequence-mode VSM Moment versus
Temperature dialog

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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**.

Figure 6-31. Data Acquisition section of the sequence-mode VSM Moment versus Temperature dialog

○   **Uniform Spacing in Temperature** (Figure 6-31) sets the VSM to take data at uniform temperature intervals.

○   **Uniform Spacing in Temperature^2** 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.

○ **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.
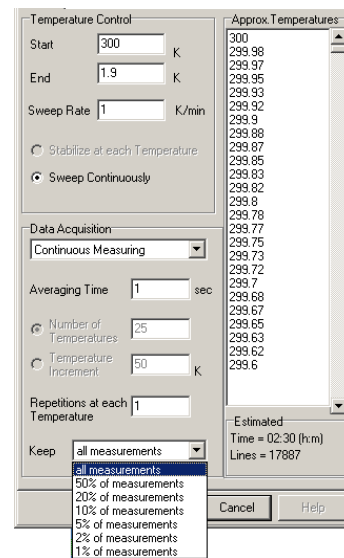
Figure 6-32. Data Acquisition section: Keep, Approximate Temperature, and Estimated settings

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Sequence-Mode VSM "Moment vs. Temp"Command

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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.
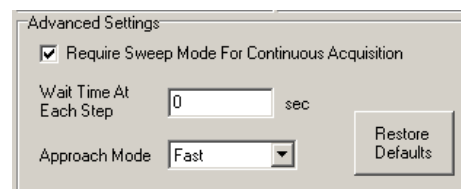
Figure 6-33. Advanced Settings section: Require sweep, Wait Time, and Approach Mode

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.
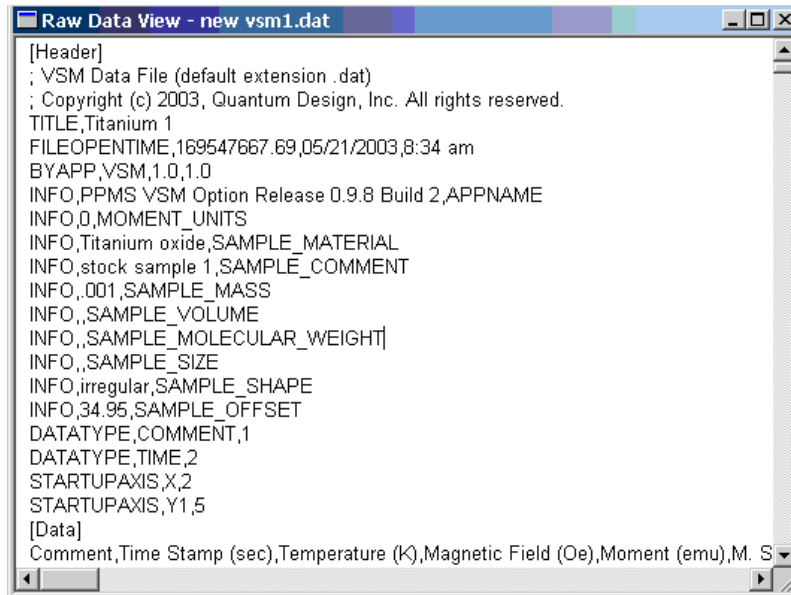
Figure 6-34. A Raw Data view of a VSM data file

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) = Asinωt
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

C H A P T E R   7

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 coilet.

- 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 chamber[1]. 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.

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[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.
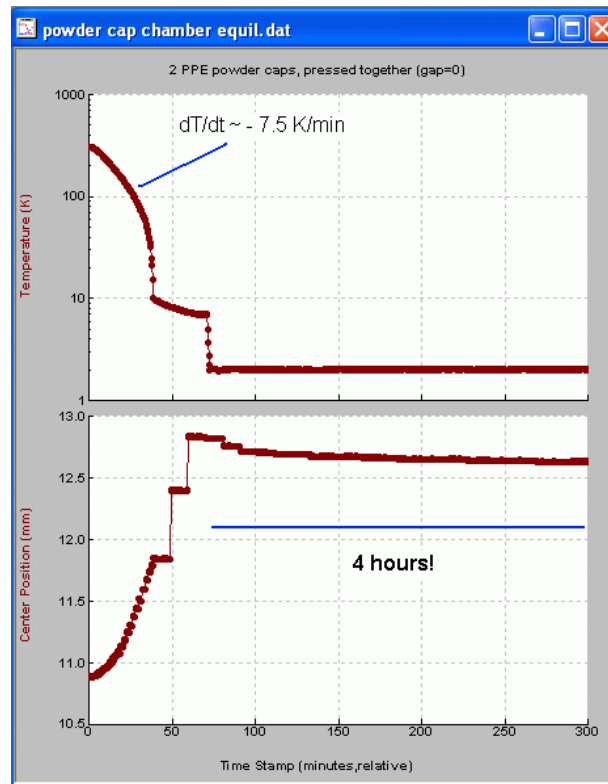
Figure 7-1. VSM data file showing change in sample position as temperature is lowered.
Note: Log temperature axis.

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 ~ 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.

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[2] in older versions of VSM software, one must plot *M.R.* instead of *Source* on the vertical axis.
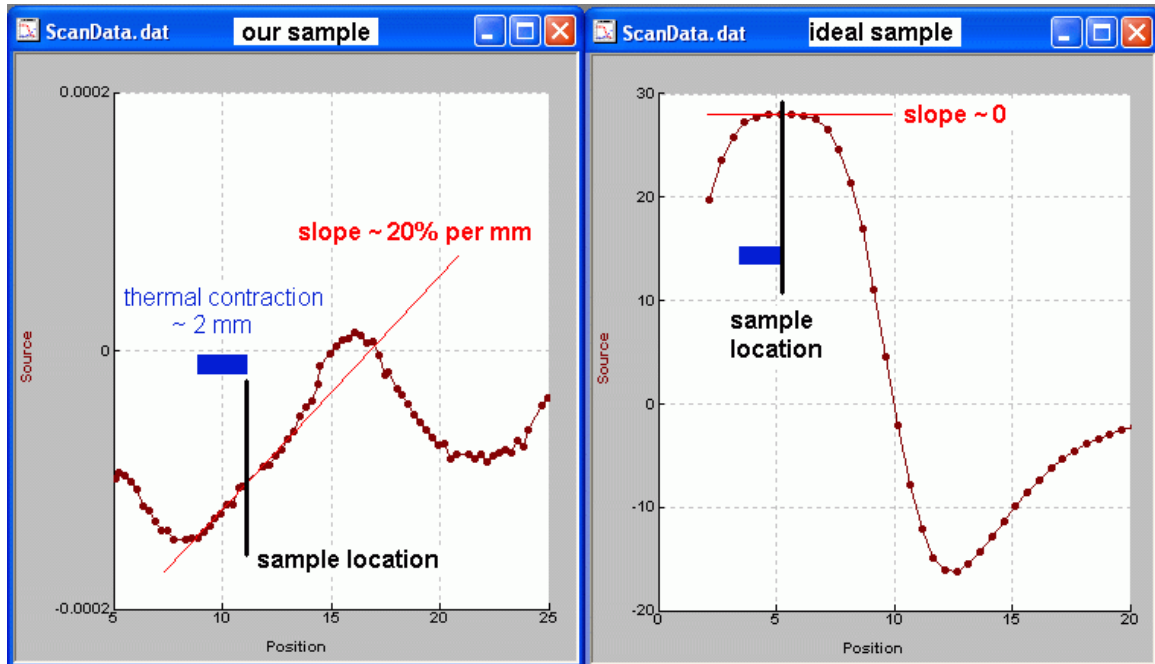
Figure 7-2. Centering scan data taken in 1 tesla applied field for the sample used in this
investigation (left) as well as an ideal sample (right). Blue bar shows position
shift expected when cooling from 300 K to 2 K without performing touchdowns.

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.
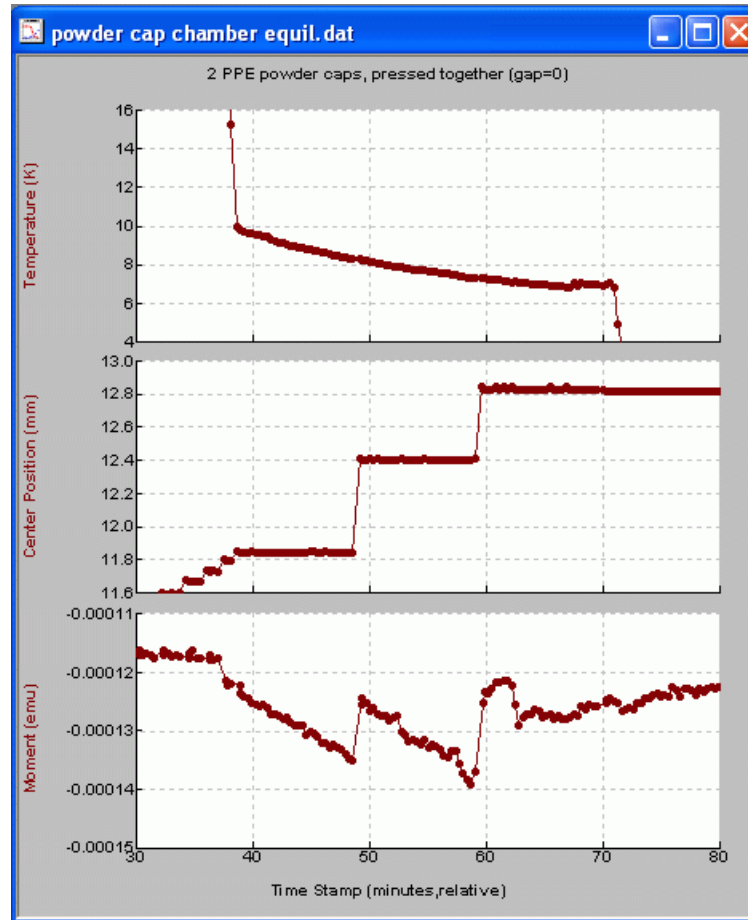
Figure7-3. Showing artifact in reported moment due to errors in vertical centering.

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, touch down 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.
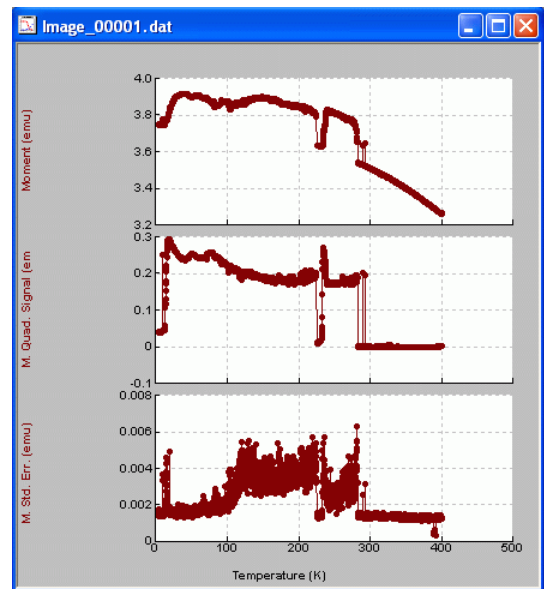

Figure 7-4. Magnetization vs. temperature data for nickel, with sample rattling in the sample holder below 290 K

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.
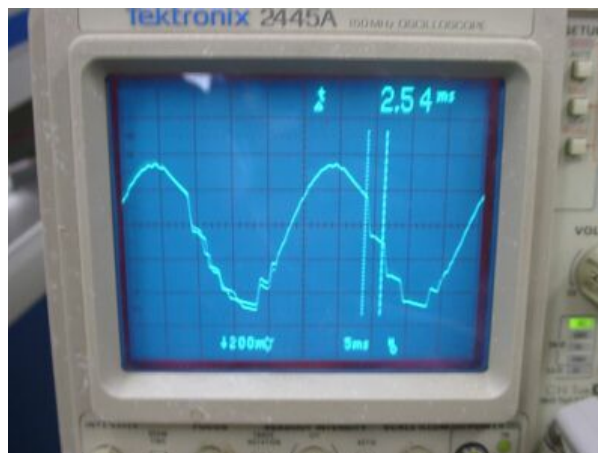
Figure 7-5. Oscilliscope trace of 40 Hz coilset signal, measured at the BNC on the VSM module. Note the jagged distortion of the signal, which would be smooth and sinusoidal for a well-mounted sample.

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 acceleration = (amplitude) * (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 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 Nb_3Sn 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 Nb_3Sn 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" Nb_3Sn 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 Nb_3Sn 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.
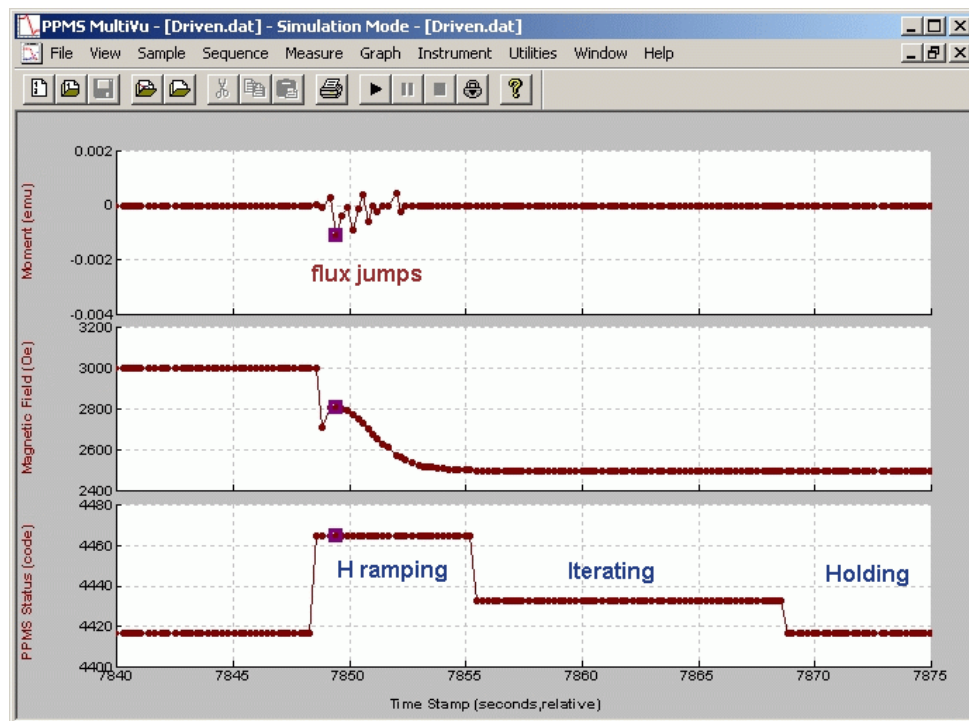
Figure 7-6. The effect of flux jumps on VSM moment versus field data during ramping and stable fields when using a 14-T magnet at fields between –2 T to +2 T.

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

A P P E N D I X   A

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.
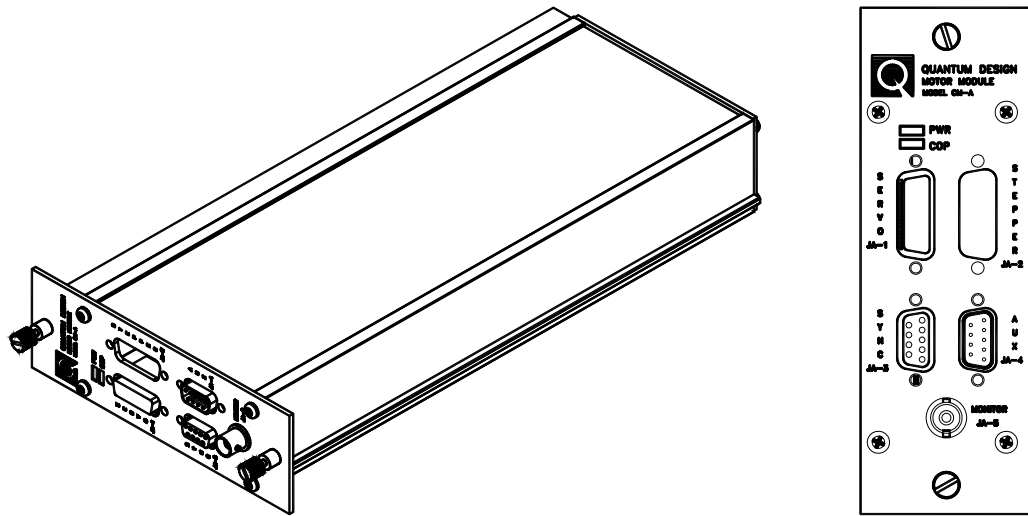
Figure A-1. Model CM-A VSM motor module (4101-100)

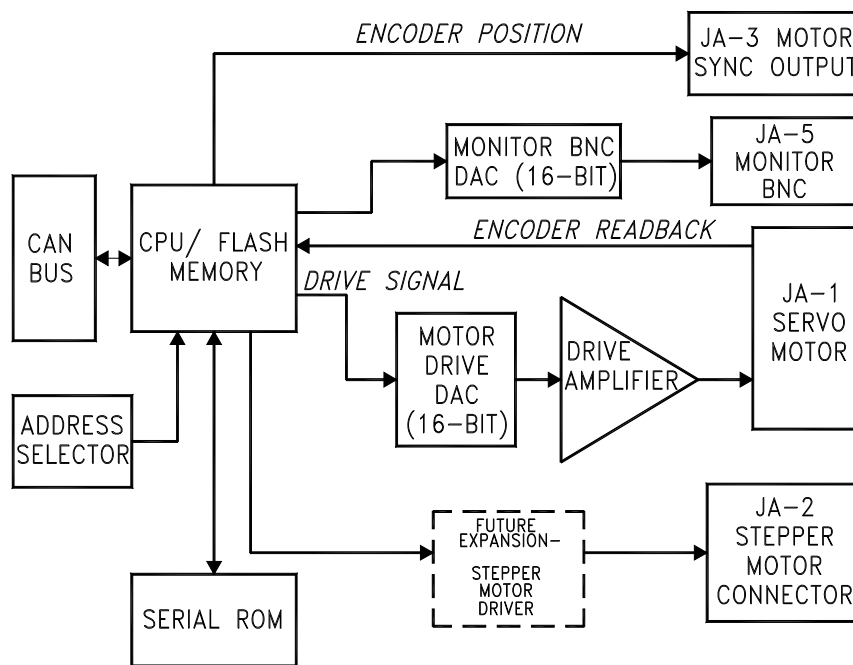## A.2.1   Functional Block Diagram

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.
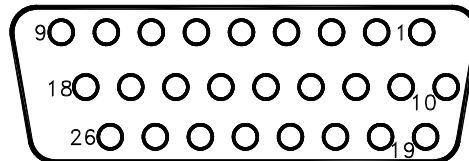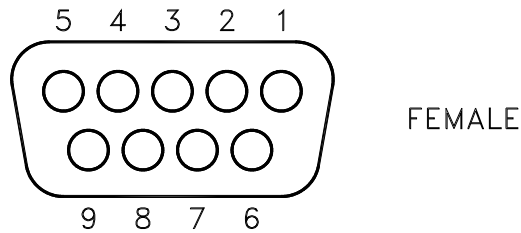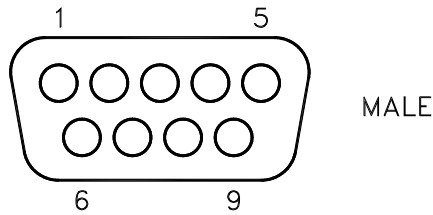

Figure A-5. JA-4: Aux connector for the Model CM-A
VSM motor module

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.
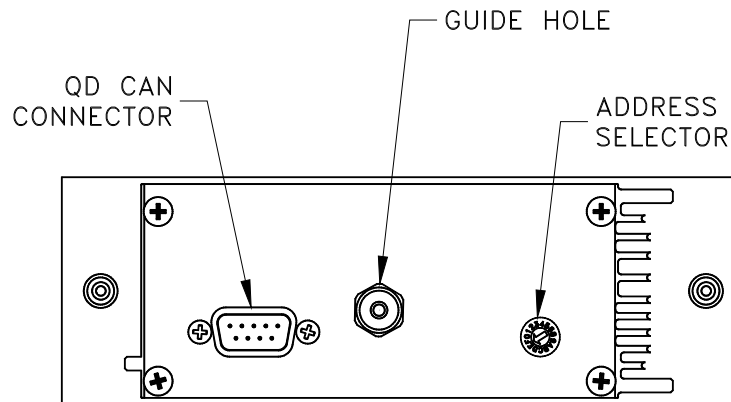

Figure A-6. Rear panel of the Model CM-A VSM motor module

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 though this connector.
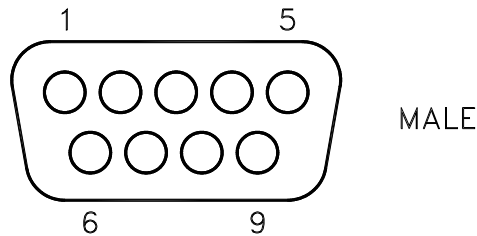

Figure A-7. Quantum Design CAN connector for the Model
CM-A VSM motor module

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:

> ⚠ **W A R N I N G !**
> 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.

A P P E N D I X   B

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.
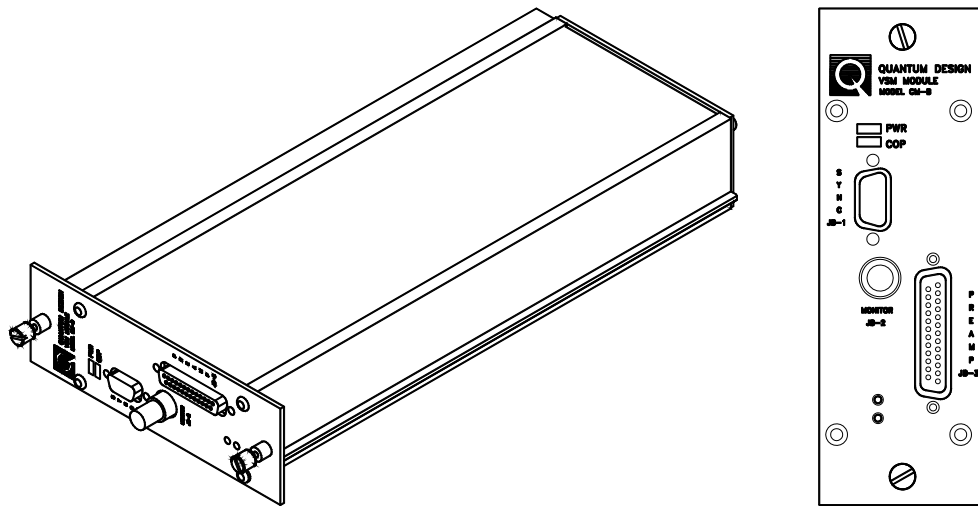
Figure B-1. Model CM-B VSM detection module (4101-150)
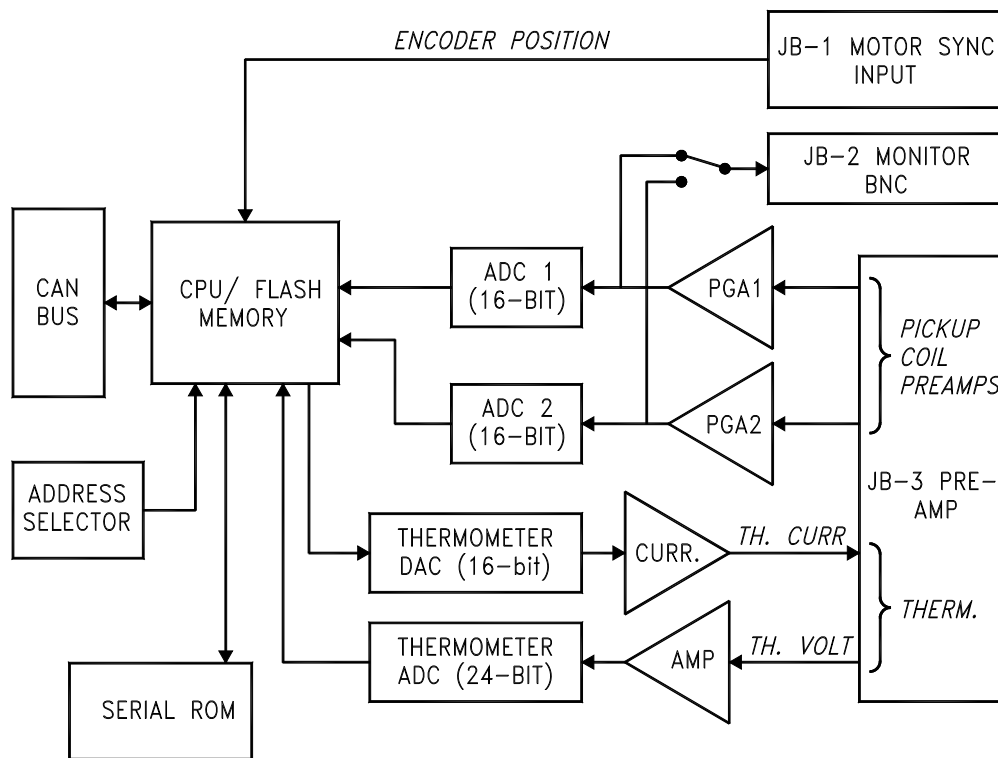
B.2.1   Functional Block Diagram

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).

```
   5   4   3   2   1
 ┌─────────────────────┐
 │  ○   ○   ○   ○   ○   │      FEMALE
 │    ○   ○   ○   ○     │
 └─────────────────────┘
      9   8   7   6
```
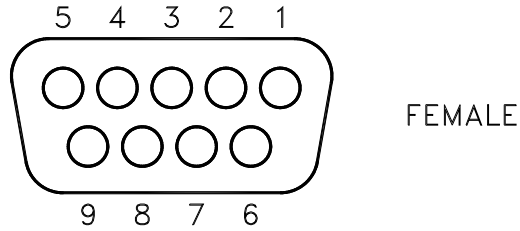
Figure B-3. JB-1: Motor sync connector for the Model CM-B
VSM detection module

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.
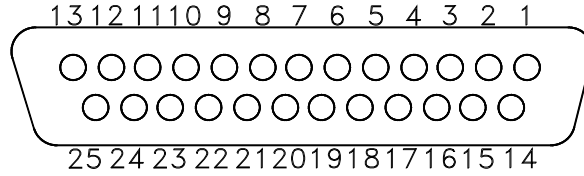

Figure B-4. JB-3: Preamp connector for the Model CM-B VSM detection module

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.
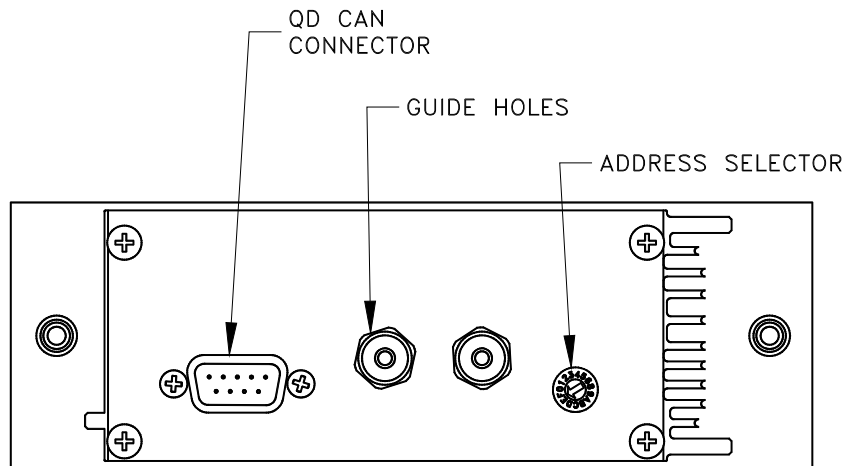

Figure B-5. Rear panel of the Model CM-B VSM detection module

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 though this connector.
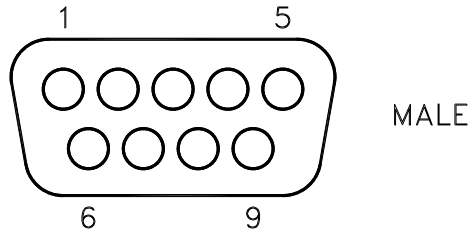

Figure B-6. QD CAN connector on rear of the Model
CM-B VSM detection module

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

A P P E N D I X   C

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.