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



Physical Property Measurement System

Hardware Manual

Part Number 1070-150, B5

Quantum Design

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U.S. Patents

- 4,791,788 Method for Obtaining Improved Temperature Regulation When Using Liquid Helium Cooling
- 4,848,093 Apparatus and Method for Regulating Temperature in a Cryogenic Test Chamber
- 5,311,125 Magnetic Property Characterization System Employing a Single Sensing Coil Arrangement to Measure AC Susceptibility and DC Moment of a Sample (patent licensed from Lakeshore)
- 5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
- 5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

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

CE

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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Contents and Conventions

P.1 Introduction

This preface contains the following information:

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

P.2 Scope of the Manual

This manual contains the information that you will need to use the Physical Property Measurement System (PPMS), including materials on its basic functionality and the hardware that is unique to it. This manual does not cover the PPMS MultiVu software, which is the Windows-based application that runs the PPMS. For detailed information about PPMS MultiVu, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual*.

The PPMS has a variety of measurement options as well as alternate set ups, so some of the manual materials might not be relevant to your equipment. For example, this manual explains how to use nitrogen-jacketed PPMS dewars. If your PPMS uses a standard dewar (i.e., one without a nitrogen jacket), you can ignore instructions that concern only liquid nitrogen. Also, this manual includes material about systems that have a magnet. If your system does not include a magnet, you can ignore those sections. All other PPMS functions are identical for systems with or without magnets.

P.3 Contents of the Manual

- Chapter 1 introduces the PPMS, including safety considerations and system setup, and explains how to contact Quantum Design.
- Chapter 2 describes and illustrates the main PPMS hardware components.
- Chapter 3 describes how the PPMS operates and gives an example of a measurement.

- Chapter 4 explains how to use and customize the PPMS and describes routine maintenance procedures such as refilling a cold dewar with helium.
- Appendix A describes and illustrates the PPMS electrical ports.
- Appendix B explains how to transfer helium and nitrogen to fill warm (empty) standard dewars and nitrogen-jacketed dewars.
- Appendix C provides maintenance instructions for the vacuum-pump assembly.

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 software.	
File >> Open	The >> symbol indicates that you select multiple, nested software options.	
STATUS	Bold text and all capital letters distinguish the names of keys located on the front panel of the Model 6000 PPMS Controller.	
.dat	The Courier font indicates file and directory names and computer code.	
<enter></enter>	Angle brackets distinguish the names of keys located on the PC keyboard.	
<alt+enter></alt+enter>	A plus sign connecting the names of two or more keys indicates keys that you press simultaneously.	
Important	Text is set off in this manner to signal essential information that is directly related to the completion of a task.	
Note	Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances.	

CAUTION!

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



WARNING!

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

WARNING!

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

Introduction and System Setup

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the function of the PPMS.
 Section 1.3 presents an overview of the system.
 Section 1.3 presents an overview of the Section 1.6 explains how to contact
- Section 1.3 presents an overview of the PPMS hardware.
- Section 1.4 covers important safety guidelines.
- Section 1.6 explains how to contact Quantum Design's service centers.

1.2 Overview of the PPMS

The Physical Property Measurement System (PPMS) provides a flexible, automated workstation that can perform a variety of experiments requiring precise thermal control. You can use the PPMS to execute magnetic, electro-transport, or thermo-electric measurements, or you can modify the system in order to perform your own laboratory experiment. The unique open architecture of the PPMS allows you to fully configure the basic PPMS platform or to use the PPMS with different PPMS measurement options, such as the AC Measurement System option, Heat Capacity option, or Ultra-Low Field option. All PPMS options, like the PPMS platform, are fully automated.

Control of the PPMS sample environment includes magnetic fields up to ± 16 T, depending on the magnet purchased, and a 1.9–400 K temperature range. Temperature is reported with a typical accuracy of $\pm 0.5\%$. Temperature can be varied with full sweep capability and slew rates from 0.01 K/min up to 12 K/min. Temperature stability is $\leq 0.2\%$ for temperatures ≤ 10 K and $\leq 0.02\%$ for temperatures > 10 K.

Please familiarize yourself with the information in this manual, which is designed to help you operate and maintain the basic PPMS platform. It is important that you understand the basic PPMS platform before you perform PPMS experiments or use PPMS options.

For information about PPMS MultiVu, which is the Windows-based software application that controls operation of the PPMS and its options, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual.*

1.3 Overview of System Hardware

Figure 1-1 illustrates a base PPMS and the approximate dimensions of each component. Note that the actual dimensions and layout of your PPMS will reflect the system that you purchase (e.g., options, type of dewar) and your laboratory. Dimensions of the electronics cabinet and the various dewars are listed in Table 1-2.

The base system includes the following hardware components, which are described in Chapter 2.

- dewar
- probe
- top-plate assembly
- probe-lifting assembly
- electronics cabinet

- Model 6000 PPMS Controller
- vacuum pump
- pumping lines
- \circ connection cables
- power cords



Figure 1-1. Components of the base PPMS and approximate dimensions (measurements are rounded¹)

¹ The inch to cm equivalences are approximate, because both measures were rounded after inches had been converted to centimeters.

Most systems also include a personal computer, a sample puck kit, and a helium-transfer kit. You can order other options to expand the capabilities of the base system, including a magnet and either the Model 6700 Magnet Controller or the Model 3120 Magnet Power Supply. The PPMS can be equipped with a 1-T, 7-T, 9-T, 14-T, or 16-T longitudinal magnet or a 7-T transverse magnet.

With the exception of 14-T and 16-T longitudinal systems and 7-T transverse systems, PPMS units can operate with one of three different types of dewars: the standard, nitrogen-jacketed, or high-capacity nitrogen-jacketed dewar. Two nitrogen fill ports located on top of the nitrogen-jacketed dewar distinguish it from the standard (non-nitrogen-jacketed) dewar. Figure 1-2 illustrates the nitrogen fill ports, and Figures 2-1–2-3 illustrate these three dewars. A liquid-nitrogen transfer adapter is included with all nitrogen-jacketed dewars.

The probe is the component that is inserted into the dewar. If the system has a longitudinal magnet, the magnet is attached to the probe, as illustrated in Figure 2-4. Any system with a magnet also includes the Model 6700 Magnet Controller or the Model 3120 Magnet Power Supply.

Please save the original packing crates for your system so that you can ship components back to Quantum Design for installation of an option, upgrading, or repair.

1.4 Safety Precautions



$W \ A \ R \ N \ I \ N \ G \ !$

The PPMS superconducting magnets produce extremely strong *three-dimensional* magnetic fields that can be dangerous and the PPMS uses cryogenic liquids for temperature control. Critical PPMS-related safety precautions include those for using superconducting magnets, for using cryogenic materials (liquids and gases), and for using electrical equipment, as is reviewed below.

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 unusual, something might be wrong with it. If so, take appropriate action.
- Supervise inexperienced users and train them in general electrical safety procedures.

The PPMS has safety features to prevent accidents from causing injury or serious equipment damage. *If you use the equipment in a manner that is not specified by Quantum Design, the protection afforded by the equipment could be impaired.*

1.4.1 Cryogens



WARNING!

Always wear protective clothing and ensure that the room has good ventilation when you work with cryogenic materials such as liquid helium and liquid nitrogen. This will protect you against cryogenic material hazards: (1) they can expand explosively when exposed to room temperature; (2) they can cause serious burns.

- Always wear protective clothing, including thermal gloves, eye protection, and covered shoes, when you work with liquid helium, liquid nitrogen, or other 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. Surfaces that have been exposed to these cryogens are extremely cold and should not be allowed to contact skin.
- 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 are generally safest.



WARNING!

Do not remove, disable, or otherwise tamper with the dewar rupture disk or the pressure-relief valves. Any type of modification can lead to dangerous operating conditions.

When liquid helium or nitrogen boils and expands in a sealed container such as the PPMS dewar, it can cause large pressure buildups. Explosions can occur if this pressure is not relieved. The PPMS dewar and probe contain pressure-relief valves and rupture disks to allow gaseous cryogens to escape before dangerous pressures are reached. Figure 1-2 illustrates these safety features.



Figure 1-2. Pressure-relief valves and rupture disk on PPMS dewar and probe head



WARNING!

If a dewar ruptures or cryogenic materials spill, vent the room immediately and evacuate all personnel.

Only perform cryogen transfers when you are in a well-ventilated room. Cryogens expand to hundreds of times their liquid volume when they boil, which occurs well below room temperature. A relatively small volume of liquid nitrogen or helium can displace all the air in a room during a spill. Nitrogen and helium are colorless and odorless, and the only symptoms of insufficient oxygen are dizziness and unconsciousness, followed by death.

1.4.2 Magnets



Never disconnect a charged magnet from the magnet control electronics (Model 6700 or Model 3120), and never disconnect any other system connections while a magnet is charged. The superconducting PPMS magnets can trap magnetic flux, so it would be possible to leave a charged magnet that is completely disconnected from the rest of the system. Under such circumstances, you have no means to discharge the magnet directly.

Several different cables have connections for magnet control. In the event that you must disconnect the probe from the magnet controller, verify that the magnet has been driven to zero field before you disconnect any cables, with the exception of the sample-chamber connection. The sample chamber connection—that is, the cable from the gray Lemo connector that is on the PPMS probe head—is the only cable that you can safely disconnect while the magnet is charged. You must leave all other connections intact.

The safety guidelines given here are generalized to a 9-T longitudinal magnet. Verify that any person wearing a pacemaker or other electrical or mechanical medical device stays at least 16.5 ft. (5 m) from the PPMS dewar. This distance applies to people located in adjacent rooms and on floors above and below the equipment, because the magnets produce *three*-

² At the current time (August 2004), 5 m should be a large enough distance to protect wearers of metallic implants or medical devices from most magnetic fields produced by Quantum Design magnets. However, the safe distance from newer magnets (in development) could be greater. Hence, personnel who work with and around the superconducting magnets should review thoroughly documentation for new equipment.

dimensional fields. The magnetic fields produced by the PPMS can be dangerous or fatal to anyone who is wearing a pacemaker or other electrical medical device. This information should be posted in the laboratory where the PPMS is operated and adjacent areas so that people wearing such devices are aware of the presence of large magnetic fields.

Important: The automated control system can turn on the magnet while the system is unattended. Furthermore, the *three-dimensional* magnetic field of the PPMS will penetrate nearby walls, the ceiling, and the floor. Therefore, your safety considerations should include such adjacent spaces. Also, note that transverse magnets produce substantially stronger fields around the dewar than longitudinal magnets do.

- The superconducting magnets supplied with PPMS units all produce strong fields that are not completely confined to the system unless it has some type of magnetic shielding. For example, PPMS superconducting magnets can disturb computer monitors, affect electron microscopes, erase credit cards, attract ferromagnetic tools, and so on. Table 1-1 summarizes some of these effects.
- ❖ Quantum Design recommends that you measure the magnetic field around the PPMS and draw a line showing where the measured field drops below 5 G. It is your responsibility to determine the location of this line, because it varies from system to system. This line is typically about 3–10 ft. (1–3 m) from the edge of the dewar. Do not bring heavy ferromagnetic objects, such as gas cylinders and large tools, within this region when the magnet is charged. Gas cylinders in the laboratory should be secured to the walls and only informed personnel should be allowed to use large tools in the presence of the PPMS. It is possible to cause injury to personnel and damage to PPMS equipment by allowing heavy objects to be attracted to the PPMS.
- Keep the helium level *above* the superconducting magnet. There is high potential for damage, such as an uncontrolled magnet quench, when the superconducting magnet is not completely covered by helium. See Sections 3.5 and 4.2.4 for more information.

1.4.3 Electricity

WARNING!

The PPMS console, personal computer, and vacuum pump are all powered by standard 120 VAC or 240 VAC power lines. 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 of its covers.
- * Keep electrical cords in good working condition, and replace frayed and damaged cords.
- ✤ Keep liquids away from the workstations.

Safe operation of the PPMS requires the appropriate electrical power input. The input power requirements for the PPMS are the following:

- 50–60 Hz, 100–120 VAC at 15 A
- 50-60 Hz, 200-240 VAC at 10 A

1.5 Environmental Considerations and PPMS Setup

You must consider a number of environmental constraints when you install the PPMS in a laboratory, including the effects of magnetic fields generated by the superconducting magnet; the physical dimensions of the equipment; vertical clearance above the dewar; and the local altitude and humidity.

The PPMS is intended for indoor use at altitudes less than about 6000 ft. (1829 m). If you will be operating the system at altitudes above our specifications, please discuss it with your Quantum Design representative. The PPMS should be operated in an ambient temperature between 5 °C and 40 °C, with a maximum relative humidity of 80% at 40 °C.

Important: The electrical safety features of the PPMS might be impaired if it is operated outside these environmental considerations.

1.5.1 Magnetic-Field Considerations

Before you install the PPMS, consider how the *three-dimensional* field generated by the superconducting magnet will affect nearby people and objects. For example, measuring out from a 9-T longitudinal magnet at full field, we obtained the surrounding radial distances at which various field strengths occur. These results are shown in Table 1-1 according to their effects on people and objects, including those located above and below the magnet. This list is not comprehensive, so you should consider *all* the equipment in your laboratory that might be affected by magnetic fields. Also, remember that a PPMS with a stronger magnet (e.g., 14 T or 16 T) will produce effects at significantly smaller distances than those listed.

ITEM	EFFECT	FIELD REQUIRED (Oe)	APPROXIMATE RADIAL ^a DISTANCE FROM THE DEWAR
Electron microscope	Disturbance	1	85.0 in. (216 cm)
Color and monochrome computer monitors (CRT type, unshielded)	Disturbance	1–5	50.0–59.8 in. (127–152 cm)
Credit cards, bank cards, etc.	Erasure	10	40.2 in. (102 cm)
Watches and micromechanical devices	Disturbance	10	40.2 in. (102 cm)
Pacemaker (lowest known field)	Disturbance	17	33.1 in. (84 cm)
Magnetic tapes	Erasure	20	31.1 in. (79 cm)
Transformers and amplifiers	Saturation	50	22.8 in. (58 cm)
Floppy disks	Erasure	350	11.8 in. (30 cm)

Table 1-1. Possible effects of the PPMS magnet, based on a system with a 9-T longitudinal magnet

^a The magnetic fields are three-dimensional, so their effects will extend above and below the magnet as well as around it.

1.5.2 Physical Dimensions

When you are deciding where you will locate the PPMS, consider the constraints below as well as the dewar and cabinet dimensions, which are listed in Table 1-2. Note that these measurements are approximate.

- Empty space is needed around the PPMS to allow regular transfers of helium and nitrogen and for a transfer vessel (usually a large portable dewar), which must be brought near the PPMS dewar.
- Vertical clearance of about 6 ft. (1.8 m)³ is needed above the dewar so that you can easily insert and extract the probe. These dimensions are suitable for inserting and removing the probe for all configurations⁴ of the PPMS dewar, including the VSM and systems that have a 14-T magnet (which requires a crane).
- Vertical clearance of about 3.3 ft. (1 m) is needed above the dewar for the sample-insertion tool and the helium-transfer line.

HARDWARE	PHYSICAL DIMENSIONS
Standard dewar (no nitrogen jacket)	45 in. high \times 19 in. diameter (114 cm ^a high \times 48 cm diameter)
Nitrogen-jacketed dewar	46 in. high \times 21 in. diameter (116 cm high \times 53 cm diameter)
High-capacity (nitrogen- jacketed) dewar	46 in. high × 28 in. diameter (116 cm high × 71 cm diameter)
EverCool dewar (w/o cold head)	45 in. high \times 22 in. diameter (114 cm high \times 55 cm diameter)
Electronics cabinet	22 in. wide \times 24 in. deep \times 45 in. high (56 cm wide \times 61 cm deep \times 114 cm high)

Table 1-2. Approximate physical dimensions of the PPMS dewars and the electronics cabinet

^a The inch to cm equivalences are approximate, because both measures were rounded after inches had been converted to centimeters.

1.5.3 Local Altitude and Humidity

The altitude and humidity of the laboratory can affect the performance of the PPMS.

The PPMS is designed to operate at altitudes below about 6000 ft. (1829 m). You can operate the PPMS at altitudes of 6000 ft. (1829 m) and above, but it is not an optimal environment. To control the sample temperature in the PPMS at temperatures that are below the boiling point of helium, the pressure difference between the inside and outside of the dewar must be monitored. If you are at a high altitude where atmospheric pressure drops significantly below 760 torr (1 atm), you might notice some problems with temperature control or some sluggishness during low-temperature operation.

³ A room with a ceiling about 10 ft. (3 m) high should provide enough vertical clearance to insert and remove the probe, depending on your setup.

⁴ These dimensions are correct as of August, 2004.

The PPMS is best suited for dry environments and it should be operated with the humidity less than 90%. Ice naturally forms when water in the air condenses and then freezes on cold surfaces, and serious system problems can be caused when even the smallest piece of ice forms inside the PPMS. As humidity increases, it is easier for water to enter the system. Eventually this will cause temperature-control problems. The PPMS does not have an airlock chamber, so the sample chamber should be warmed to room temperature and vented continuously with a clean, dry gas whenever the chamber is opened to the atmosphere. The system is designed so that you can warm and vent the sample chamber by pushing a few buttons.

1.6 Contacting Quantum Design

If you have questions or problems related to your QD equipment, please contact your local QD service representative at one of the offices listed below. When you call, please be able to give the representative a full description of the problem, including the circumstances involved and the recent history of your system.

United States

Quantum Design World Headquarters 6325 Lusk Boulevard San Diego, CA 92121

Tel: 1-858-481-4400 1-800-289-6996 Fax: 1-858-481-7410

Email: <u>service@qdusa.com</u> Web: http://www.qdusa.com

Service for Canada, Mexico, the U.S., and other countries not listed below

Europe

L.O.T.—Gmbh & Co KG Im Tiefen See 58 D-64293 Darmstadt, Germany

Tel: 49-6151-880631 Fax: 49-6151-896667

Email: <u>qd.euroservice@lot-oriel.de</u> Web: <u>http://www.lot-oriel.com</u>

Service for Austria, Belgium, Crete, Croatia, Czech Republic, Denmark, England, Finland, France, Germany, Greece, Hungary, Ireland, Israel, Italy, Luxembourg, Netherlands, Norway, Poland, Portugal, Russia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey, and Yugoslavia

Japan

Quantum Design Japan Sanpo Ikebukuro Building Annex 4-32-8 Ikebukuro Toshima-ku, Tokyo 171-0014, Japan

Tel: 81-3-5954-8570 Fax: 81-3-5954-6570

Email:qdjapan@tkb.att.ne.jpWeb:http://www.qd-japan.com

Service for Japan

Korea

Quantum Design Korea Kyungbin Building, Fourth Floor 517-18 Dogok-dong, Kangnam-gu Seoul, 135-270, Korea

Tel: 82-2-2057-2710 Fax: 82-2-2057-2712

Web: <u>http://www.qdkorea.com</u>

Service for Korea

People's Republic of China

Oxford Instruments Beijing Office Room 714, Office Tower 3 Henderson Center No. 18 Jianguomennei Ave Dongcheng District Beijing 100005 P.R. China

Tel: 8610-6518-8160/8161/8162 Fax: 8610-6518-8155

Email:lambert@oxford-instruments.com.cnWeb:http://www.oxford-instruments.com.cn

Service for People's Republic of China

Taiwan

Omega Scientific Taiwan Ltd. 5F-1, No. 415, Sec. 4 Hsin Yi Road Taipei, Taiwan R.O.C.

Tel: 886-2-8780-5228 Fax: 886-2-8780-5225

Email: lonson.lin@omega-cana.com.tw

Service for Taiwan, Hong Kong, Singapore

Hardware

2.1 Introduction

This chapter contains the following information:

- Section 2.2 describes the dewar, including its functions and the three main types.
- Section 2.3 describes the probe and its main components.
- Section 2.4 describes the top-plate assembly.
- Section 2.5 describes the probe-lifting assembly.
- Section 2.6 describes the Model 6000 PPMS Controller.

- Section 2.7 describes the optional Model 6700 Magnet Controller.
- Section 2.8 describes the electronics cabinet.
- Section 2.9 describes the vacuum pump.
- Section 2.10 describes the sample puck and the puck tools.

2.2 Dewar

The dewar contains the liquid-helium bath in which the probe is immersed. Primarily constructed of aluminum, the outer layer of the dewar has reflective superinsulation to help minimize helium consumption. The outer layer is evacuated through a valve on the top of the dewar (Figures 2-1, 2-2, and 2-3)—this evacuation valve *must not* be modified or altered.¹ The dewar regions that are evacuated contain activated charcoal on cold surfaces to aid cryopumping.

Most PPMS units can operate with one of three different types of dewars: the standard dewar (Figure 2-1), the nitrogen-jacketed dewar (Figure 2-2), or the high-capacity nitrogen-jacketed dewar (Figure 2-3). The exceptions include EverCool units and systems that have a 14-T or 16-T longitudinal magnet or a 7-T transverse magnet. Instructions for filling warm (or empty) dewars with nitrogen and helium are given in Appendix B, and instructions for refilling cold dewars with nitrogen and helium are given in Sections 4.7.2.

Note: Due to physical constraints, EverCool systems and systems with a 14-T or 16-T longitudinal magnet or a 7-T transverse magnet have special dewars that are not discussed in this manual. For more information about EverCool dewars, refer to the *PPMS EverCool Dewar Option User's Manual*. Contact Customer Service at Quantum Design if you have questions about the other dewars.

¹ If you have any questions about this issue, please contact Customer Service at Quantum Design.

2.2.1.1 STANDARD DEWAR

Figure 2-1 illustrates a top view and a cross-section of a standard dewar. The standard dewar contains a set of heat shields around the neck of the helium container and does not have a nitrogen jacket. Otherwise, it is similar to but slightly smaller than the nitrogen-jacketed dewar.

Standard dewars have a 30 L liquid-helium capacity.



Figure 2-1. Top view and cross-section of a standard dewar, illustrating construction of dewars without a nitrogen jacket (dimensions are in inches)

2.2.1.2 NITROGEN-JACKETED DEWAR

Nitrogen-jacketed dewars (Figure 2-2) consume significantly less liquid helium than dewars without nitrogen jackets. Two liquid-nitrogen-fill ports give the top of the nitrogen-jacketed dewar a distinctive appearance.

The operating efficiency of nitrogen-jacketed dewars is partly due to a layer of liquid nitrogen sandwiched between the superinsulation and the liquid helium, which further insulates the helium bath, as does the vacuum in the region between the liquid helium and the liquid nitrogen.

A liquid-nitrogen transfer adapter is included with all nitrogen-jacketed dewars. The transfer adapter (Figure 2-2) is a short, L-shaped tube that fits on the end of most standard liquid-nitrogen transfer lines and facilitates the liquid-nitrogen fill procedure.

The standard nitrogen-jacketed dewar has a 30 L liquid-helium capacity and a 40 L liquidnitrogen capacity.



Figure 2-2. Top view and cross-section of a nitrogen-jacketed dewar and a side view of a liquid nitrogen transfer adapter (dimensions are in inches)

2.2.1.3 HIGH-CAPACITY NITROGEN-JACKETED DEWAR

High-capacity nitrogen-jacketed dewars are used solely with systems that have a 7-T or 9-T longitudinal magnet. Figure 2-3 illustrates a top view and a cross-section of a high-capacity nitrogen-jacketed dewar, which has a 87 L liquid-helium capacity and a 48 L liquid-nitrogen capacity.



Figure 2-3. Top view and cross-section of a high-capacity nitrogen-jacketed dewar (dimensions are in inches)

2.3 Probe

The probe (Figure 2-4) is immersed in the liquid-helium bath inside the dewar. A sophisticated device with delicate components, the probe incorporates the basic temperature-control hardware, the superconducting magnet, the helium-level meter, the gas lines, the sample puck connectors, and various electrical connections. You can prevent damage to the probe by following the probehandling instructions in Section 4.2.1.

The probe is composed of several concentric stainless steel tubes and other important elements. Its outer layer isolates the sample chamber from the liquid-helium bath. Two concentric tubes, separated by a sealed, evacuated region, prevent heat exchange between the sample chamber and the helium bath. The vacuum space between the outer and inner vacuum tubes contains reflective superinsulation to minimize radiative power loss into the helium bath. An aluminum heat shield in the vacuum region directs heat to the neck of the dewar, where there is no liquid helium. A metal bellows at the bottom of the probe prevents it from being damaged by differential thermal expansion between the outer vacuum tube and the heat shield. A cap at the bottom of the probe protects the bellows. The protective cap is not sealed, and liquid helium flows freely into it.

Major components of the probe are the sample chamber, impedance assembly, optional magnet, baffled rods, and probe head, which are discussed in the following sections.



Figure 2-4. Major components of the PPMS probe

2.3.1 Sample Chamber

The sample chamber is inside the two vacuum tubes. The lower 3.9 in. (10 cm) of the sample chamber is constructed of copper in order to provide a region of uniform temperature. The very base of the sample chamber contains a 12-pin connector that contacts the bottom of an installed sample puck. Two thermometers and a heater are immediately below the sample puck connector. Their proximity to the copper sample puck and mating connector helps them maintain close thermal contact with the puck and sample during experiments. The wiring for the sample puck connections, heaters, and thermometers runs up the outside of the sample chamber to the probe head. The pins from the sample puck connector are wired to the pins on the gray-ringed Lemo connector on the probe head. Appendix A has a list of pinouts for the sample puck, sample puck connector, and gray Lemo connector.

The region between the sample chamber and the inner vacuum tube is referred to as the cooling annulus. Helium is pulled through the impedance tube into the cooling annulus so that it can warm and cool the sample chamber evenly.

2.3.2 Impedance Assembly

The impedance assembly enables and disables the flow of helium into the cooling annulus from the dewar. The assembly consists of a narrow tube (the impedance), a heater that warms the impedance, and a thermometer that indicates when the impedance is warm. When the impedance is warm, a bubble forms inside the tube, blocking the flow of liquid helium. Then when the impedance heater is off, the liquid helium cools the impedance tube and flows into the cooling annulus, where it either vaporizes or fills the annulus, depending on the pressure inside the annulus. The cap at the bottom of the probe protects the impedance tube.

Newer probes (those manufactured since January 1998) are enabled for the Continuous Low-Temperature Control (CLTC) option—they have a carefully tuned second impedance in parallel with the primary impedance. Owners of earlier model probes can purchase the CLTC option by contacting Quantum Design. See Sections 3.3 and 4.3 for more information on temperaturecontrol modes.

2.3.3 Baffled Rods

The rods that run the length of the probe contain electrical connections to the magnet and impedance assembly. One of the rods contains the helium-level meter. Several baffles provide support for the rods. The rods are delicate and cannot support the full weight of the probe.

2.3.4 Probe Head

The top part of the probe that protrudes out of the dewar is referred to as the "probe head." The probe head contains the two helium-fill ports and all the connection ports for attaching gas, vacuum, and electrical lines from the Model 6000 PPMS Controller. Most of the ports and connections are on the back of the probe head. Figures 2-4 and A-3 illustrate the probe head, and Appendix A discusses the ports and connections.

The probe head includes the access port into the sample chamber. A blank flange covers the access port unless certain PPMS options have been installed.

2.3.5 **Optional Magnet**

The PPMS can be purchased with a 1-T, 7-T, 9-T, 14-T, or 16-T longitudinal magnet or a 7-T transverse magnet. The magnet is a superconducting solenoid composed of a niobium–titanium alloy embedded in copper. It is on the outside of the probe, so it is always immersed in liquid helium.

The magnet coil constitutes a closed superconducting circuit. The persistence switch is a small heater on the magnet wire that drives a section of the magnet non-superconducting. The persistence switch allows the magnet controller (either the Model 6700 or the Model 3120) to be switched into the magnet circuit so that the magnetic field can be changed. When the heater is turned off, the entire magnet can superconduct, which eliminates the need for a current source during constant field operation. This state is referred to as the **Persistent** mode of the magnet. Section 3.5 discusses magnetic-field control in more detail.

For 1-T, 7-T, 9-T, 14-T, and 16-T longitudinal magnets, the magnetic field is centered 2.1 in. (5.4 cm) above the surface of an installed sample puck, but the field uniformity varies. The 7-T transverse magnets are shipped with the center of the field 1.6 in. (4.0 cm) above the surface of an installed puck.

2.4 **Top-Plate Assembly**

The top-plate assembly (Figure 2-5) consists of the components that seal the sample chamber: the top plate; a centering ring, O-ring, and hinge clamp; the top-plate baffle assembly; and a threaded adapter.

The top plate is a KF blank flange that closes the sample-chamber access port. A centering ring with an O-ring around its diameter fits between the blank flange and the access port in order to seal the sample chamber. A hinge clamp holds the blank flange on top of the access port.

The top-plate baffle assembly is a set of baffles on a light G-10 rod that is attached to the bottom of the blank flange. The baffles confine thermal gradients to specific regions of the sample chamber, aiding thermal control so that the system can achieve temperatures as low as 1.9 K. A small threaded adapter at the end of the baffle rod allows you to attach other components to the rod. Note that some PPMS options seal the sample chamber differently and do not use the components shown in Figure 2-5.



Figure 2-5. Cross-section of top-plate assembly

2.5 **Probe-Lifting Assembly**

The probe-lifting assembly (Figure 2-6) is used to install and remove the probe. The assembly consists of a U-bolt, a locking mechanism, and a plate. The plate fits just underneath the sample-chamber access port, which is located on the probe head (see Figures 2-4 and 2-5).

You typically will use the probe-lifting assembly in conjunction with a hoist. You can tie or pass a rope through both the U-bolt and a fixture in the ceiling so that you can slowly raise and lower the probe. When the probe-lifting assembly is not being used, remove it from the probe. Section 4.7.4 explains how to use the probe-lifting assembly.



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Figure 2-6. Probe-lifting assembly
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2.6 Model 6000 PPMS Controller

The Model 6000 PPMS Controller (Figure 2-7) is an integrated user interface that houses the electronics and the gas-control valves for the PPMS. The Model 6000 contains the CPU board, motherboard, and system bridgeboard. The CPU board is the system processor, the motherboard controls system integration, and the system bridgeboard supplies temperature readings. Gas valves and gas lines inside the Model 6000 are used to control temperature.

Refer to Sections 2.6.1.1 and 2.6.1.2 and Appendix A for more information on the Model 6000. Refer to the *Physical Property Measurement System Firmware Manual* and the *Physical Property Measurement System Commands Manual* for practical information about using the Model 6000.



Figure 2-7. Front panel of Model 6000 PPMS Controller

2.6.1.1 MODEL 6000 FRONT PANEL

Figure 2-7 illustrates the front panel of the Model 6000, which has a power button, a display screen, a contrast knob, a number pad, and a signal output (BNC connector), as well as menu keys, arrow keys, and two status LEDs.

The status LEDs light up during remote control of the system and when an error is logged into the data file.

The signal output comes from an internal digital-to-analog converter. The signal output can be linked to any one of about 30 system parameters, including temperature, field, position, excitation current, and resistance. The output has a -10 V to +10 V range, and values for the linked parameter must be specified for both 0 V and +10 V. This analog output is linked in parallel to the "A1" analog output port on the Model 6000 rear panel.

2.6.1.2 MODEL 6000 REAR PANEL

The rear panel of the Model 6000 has ports for all system connections, including necessary PPMS connections, connections to optional Quantum Design hardware, and auxiliary connections that accommodate interfacing with other devices. Appendix A discusses these ports in more detail.

2.7 Optional Magnet Controller

A PPMS with a magnet will also include a magnet controller: either the Model 6700 or the Model 3120.

If you ever have a magnet-related question or encounter a problem when you are using a magnet, contact Customer Service at Quantum Design.

2.7.1 Model 6700 Magnet Controller

CAUTION!

Do not alter or remove the connection from the Model 6700 to the "P7–Magnet" port on the Model 6000. Altering or removing this connection could destroy the 24 V power supply in the Model 6000 and/or Model 6700 and void the manufacture's warranty.

The Model 6700 Magnet Controller (Figure 2-8) is a bipolar power supply that allows smoother ramping through zero (0) field than traditional one-sided power supplies. As is discussed in Section 3.5, the Model 6700 uses four different approach modes to control charging and discharging of the magnet.

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0		o
	MODEL 6700 MAGNET CONTROLLER	

Figure 2-8. Front panel of the Model 6700 Magnet Controller

2.7.1.1 MODEL 6700 MAGNET CONTROLLER FRONT PANEL

There are three LEDs on the front panel of the Model 6700: the "Logic Power" LED, which is lit when the Model 6000 is powered on, and the "DC Enable" and "On-Line" LEDs, which are lit whenever the system is deliberately charging or discharging the magnet.

2.7.1.2 MODEL 6700 MAGNET CONTROLLER REAR PANEL

The rear panel of the Model 6700 has a power switch that (indirectly) turns off the current to the magnet. *Never* turn off the power to the Model 6700 while the magnet is ramping.

CAUTION!

Never turn off the power to the Model 6700 while the magnet is ramping.

2.7.2 Model 3120 Magnet Power Supply

See Model 3120 Magnet Power Supply user's manual for more information

2.8 Electronics Cabinet

The electronics cabinet (Figure 2-9) holds the Model 6000 and Model 6700 if present, the vacuum pump, and a power strip. It also has room for the additional hardware and electronics needed with some PPMS options. Refer to Section 1.4.3 for electrical specifications for the cabinet.



Figure 2-9. PPMS electronics cabinet

2.9 Vacuum Pump

A direct-drive pump operates continuously to control pressure in the sample chamber and to aid thermal control. Valves in the Model 6000 regulate the vacuum and the gas-flow rates. Figure 2-10 shows one of the models of pumps used in the PPMS.

The pump is installed in the bottom of the electronics cabinet. It should have an oil-mist filter attached to its exhaust line and a foreline trap on its input to protect the system from contamination. The oil-mist filter is located on an inside wall of the electronics cabinet.

The vacuum pump must be maintained to ensure optimal performance of your PPMS. Pump operation and maintenance are covered in Section 4.7.3 and Appendix C.



Figure 2-10. An oil-mist direct-drive pump used to control pressure and temperature in the PPMS

2.10 Sample Puck and Assorted Tools

The sample puck (Figure 2-11) is a unique modular component that gives the PPMS great flexibility. The puck holds the sample for many experiments that use the base PPMS platform and that do not require motion of the sample. Some options, such as the AC Measurement System (ACMS) option and the horizontal rotator options, do not use the sample puck.



Figure 2-11. Top and bottom views of a sample puck

The puck is a 0.91 in. (2.3 cm) diameter disk that is constructed of oxygen-free high-conductivity copper that maintains high thermal uniformity. It has been gold-plated to prevent oxidation. The system thermometers and heaters are located directly beneath the installed puck, so temperature control is intimately related to the temperature of the sample. Options that use other sample-mounting techniques often have an additional thermometer located near the sample.

The base of the puck contains 12 solder pads through which electrical leads establish contact with the sample (you supply these leads). These solder pads are hard-wired to a set of 12 pins on the base of the puck. When you install the puck, the 12 pins connect to the sample-puck connectors located on the bottom of the sample chamber and then ultimately to the pins of the gray-ringed Lemo connector on the probe head. The puck is keyed to ensure that the electrical connectors align properly.

The last solder pad (counting clockwise) is square instead of round. You will use this solder pad as a reference point to help you wire sample leads to the proper solder pads. Refer to Section 4.4 for sample-mounting instructions.

Each PPMS option includes several pucks, so you can mount different samples on different pucks. You also can configure each puck for a different type of experiment.

2.10.1 Puck-Insertion Tool

The puck-insertion $tool^2$ is a long rod used for installing the puck in the sample space. The lever of the puck-insertion tool is engaged when it is lying flat across the handle, as is shown in Figure 2-12. When the lever is engaged, the tool grips the puck by a groove in its outer rim.





The puck-insertion procedure is described in Section 4.5. The procedure is easy and requires only a few seconds after you have warmed the sample chamber to room temperature.

2.10.2 Puck-Adjustment Tool

The puck-adjustment tool (Figure 2-13) is used to adjust the tension in the fingers of the puck so that the fingers maintain solid thermal contact with the 12-pin connector located at the bottom of the sample chamber.

The puck-adjustment tool consists of two metal cylinders. In Figure 2-13, Cylinder 1 is the finger spreader. Cylinder 2 is the finger contractor and the test cutout. The finger spreader and the finger contractor adjust the tension of the chuck fingers. The test cutout, which has the same dimensions as the cutout in the heater block, tests how well the chuck fingers will contact the heater block.

² The puck-insertion tool is also called the puck-extraction tool or sample-insertion tool, depending on context.


Figure 2-13. Puck-adjustment tool

You will use the puck-adjustment tool after you have inserted the sample puck into the sample chamber approximately 10 times or whenever the puck fits loosely into the bottom of the chamber. Instructions for using the puck-adjustment tool are given in Section 4.7.1.

2.10.3 Puck-Wiring Test Station

The puck-wiring test station³ (Figure 2-14) is used to verify the contact between a sample and puck. The test station contains three sets of contacts, all wired in series: a Lemo connector identical to the sample-chamber connector on the probe head, a puck connector, and 12 banana jacks. PPMS measurement options (e.g., Heat Capacity) come with templates that allow you to label the banana plug functions when the standard cabling is being used for an option.



Figure 2-14. Puck-wiring test station

³The puck-wiring test station is also referred to as the P150 sample-wiring test station.

Theory of Operation

3.1 Introduction

This chapter contains the following information:

- Section 3.2 illustrates the main PPMS subsystems and their interactions.
- Section 3.3 describes temperature control and operational regimes.
- Section 3.4 describes atmospheric control and gas-line configuration.
- Section 3.5 describes magnetic-field control.
- Section 3.6 describes the helium-level meter and monitoring helium levels.
- Section 3.7 describes features of the Model 6000 that can be customized.
- Section 3.8 presents an example of a measurement.
- Section 3.9 describes variables to consider during a measurement.

3.2 PPMS System Block Diagram

Figure 3-1 illustrates how the temperature control, gas-flow control, magnetic-field control, and helium-level metering subsystems are incorporated in the PPMS.



Figure 3-1. PPMS block diagram (eliminate in the picture reference to Model 6700)

3.3 Temperature Control

Figure 3-2 shows cross sections of the PPMS probe, including the components that control temperature. The outer layer of the probe is an evacuated region filled with reflective superinsulation. This layer is between the liquid-helium bath and cooling annulus, where it minimizes thermal exchange between the sample chamber and the 4.2 K liquid-helium bath. It contains an aluminum heat shield that directs heat to the neck of the probe rather than into the helium bath, where it would increase the rate of helium consumption. Without this evacuated region, temperature control would be difficult or impossible and helium consumption would be significantly higher.

The cooling annulus is the active region of temperature control. The continuously pumping vacuum pump draws helium from the dewar through the impedance tube and into the cooling annulus. The helium vapor flows through the annulus at rates that are controlled by a flow-control valve in the Model 6000 PPMS Controller.

The sample chamber is also usually kept at a pressure of a few torr with helium gas so that the walls of the sample space can maintain thermal contact with the sample. The sample chamber has a top-plate baffle assembly that helps isolate the sample space at the bottom of the chamber from the heat radiated by room-temperature components at the top. This baffle assembly is required for the sample space to reach the lowest attainable temperatures.

Sample temperature is monitored by a platinum resistance thermometer and a negative temperature coefficient (NTC) thermometer that are mounted directly beneath the electrical connectors for the sample puck. The platinum thermometer reads temperatures ranging between approximately 80 K and 400 K; the NTC thermometer reads temperatures ranging between approximately 1.9 and 100 K. A weighted average of the two thermometer readings is used in the crossover region between 80 K and 100 K. Another NTC thermometer, which is not shown in Figure 3-2, is mounted just above the sample space to monitor the temperature gradients in the chamber.

3.3.1 Temperature-Control Modes

The PPMS offers three unique operational regimes for controlling temperature in the sample space—one for high temperatures and two for low temperatures. The high-temperature regime is used for temperatures above the liquid-helium boiling point (4.2 K at 1 atm), which is the so-called "crossover temperature." The low-temperature regimes, Continuous Low-Temperature Control (CLTC) and pot-fill mode, are used to regulate temperatures below the crossover temperature regime can be used to lower the sample-space temperature to about 1.9 K, but their characteristics and advantages differ, as shown in Table 3-1.

Since January 1998, PPMS systems have included the high-temperature regime and both low-temperature regimes, with CLTC shipped as the default mode. Before that time, the PPMS included only the high-temperature regime and pot-fill mode, but owners of such systems can add the CLTC option as a purchased upgrade.



Figure 3-2. Cross-sections of the PPMS probe and its temperature-control components

3.3.1.1 HIGH-TEMPERATURE CONTROL

At temperatures above about 4.2 K, the system cools the sample space by drawing cold helium vapor, at a variable rate, through the impedance tube into the cooling annulus and across the outside of the sample chamber. Even when the sample space is not being cooled, the system maintains a helium flow of about 100 cc/min through the cooling annulus.

A block heater is mounted at the base of the sample chamber (see Figure 3-2), where it heats the sample to the desired temperature and warms the vapor in the cooling annulus, thus uniformly warming the entire sample space. Thermal gradients in the sample space are further minimized by a neck heater, which is wrapped around the sample chamber and located just above the sample space and near the neck thermometer.

The flow-control valve and the block and neck heaters use the sample temperature and neck temperature as feedback to obtain rapid thermal control. Maximum warming and cooling rates are around 6 K/min.

3.3.1.2 CONTINUOUS LOW-TEMPERATURE CONTROL

The system uses the Continuous Low-Temperature Control (CLTC) option to regulate temperatures below about 4.2 K by drawing cold helium gas through the carefully tuned CLTC flow impedance that restricts the gas flow. Flow through the primary impedance is completely turned off and the helium gas is drawn from the CLTC impedance through the annulus to cool the sample space. The system heaters warm the gas and the sample space directly.

CLTC mode includes a precooling phase that begins when the temperature in the sample space is about 11 K. The precooling minimizes thermal gradients in the sample chamber so that the unit can indefinitely maintain temperatures below 4.2 K and ensures that the temperature of the chamber smoothly transitions through 4.2 K. The precooling method uses aggressive feedback on multiple parameters. This might cause a temporary loss of temperature control, reflecting processes that prevent liquid helium from collecting in the annulus while the neck is cooled. However, the duration of each precooling process is heavily dependent on the thermal history of the system.¹

3.3.1.3 POT-FILL MODE TEMPERATURE CONTROL

In pot-fill mode, the system initiates a pot fill at about 4.2 K when it fills the cooling annulus with a controlled amount of liquid helium and manipulates the boiling point of the helium. The liquid helium is drawn through the primary impedance tube, with the impedance heater off. The fill procedure is regulated by the pressure difference between the cooling annulus and the dewar. When the annulus is almost full, which takes about 45 minutes, the impedance heater is turned on, warming the impedance tube until the helium pressure inside the tube prevents liquid helium from entering either end. This state is commonly called "on the pot."

The system can use pot-fill mode to maintain temperatures of about 1.9 K for hours.² The liquidhelium bath around the sample space provides a uniform, stable thermal environment. However, it can be difficult to maintain a temperature very close to the boiling point of the liquid helium because the control mechanisms in the high-temperature and pot-fill modes are so different.

When the system is "on the pot," it controls temperature increases and decreases by opening and closing the flow-control valve in the Model 6000 and by using the heaters. For cooling, the

¹ For example, a rapid change from room temperature to 2 K will cause wild temperature oscillations for some time, while a slow change might require little-to-no precooling.

 $^{^{2}}$ The length of time is impossible to estimate because it depends on the equipment and the experimental situation, but the minimum will probably be 3–4 hours.

system opens the flow-control valve, which decreases the pressure above the liquid helium, thereby lowering the boiling point of the helium. The temperature of the liquid helium in the annulus drops accordingly. For warming, the system closes the valve slightly, allowing the pressure in the annulus to increase, and subsequently raising the boiling point of the helium. The heaters are used for short time periods to accelerate the warming process.

In the event you reset the temperature from below 4.2 K to above 4.2 K, it will take about 45 minutes to empty the cooling annulus. During this time, the system cannot control temperature in the sample space.

3.3.1.4 SELECTING THE LOW-TEMPERATURE CONTROL MODE

CLTC is the default mode of low-temperature control in the PPMS, but you can use the mode that best meets your experimental needs. Table 3-1 summarizes the general characteristics and advantages of each mode. Low-temperature control modes are changed by using the MultiVu software application or the Mon 6000 utility, as explained in Chapter 4, "System Operation." For more information on MultiVu and the Mon 6000 utility, see the *Physical Property Measurement MultiVu Application User's Manual* and the *Physical Property Measurement System Firmware Manual*, respectively.

Note: Because the two control modes are so different, sometimes you can use pot-fill mode to cool the chamber when CLTC mode is not bringing temperatures below 4.2 K. Rather than stopping the experiment to investigate the cooling problem, you can switch to pot-fill mode and attempt to bring the unit to your target temperature. If the unit cools successfully when you use pot fill, you might be able to complete the experiment before you fix the cooling problem.³

CONTROL METHOD	CHARACTERISTICS	ADVANTAGES	DISADVANTAGES
CTLC	 Begins a precooling phase at about 10 K Reaches low temperatures without collecting liquid helium around the sample space 	 Transitions through 4.2 K helium boiling point smoothly Attains stable temperatures at (and near) 4.2 K helium boiling point Increases temperature quickly and smoothly 	 Control of temperature might be temporarily lost at beginning of precooling phase, depending on thermal history Control of temperature is relatively less stable than with pot-fill mode
Pot-fill mode	 Takes over temperature control at about 4.2 K Fills cooling annulus with liquid helium and manipulates boiling point of helium 	 Maintains extremely uniform thermal environment for the sample space Maintains a very quiet thermal environment for the sample space Provides best absolute temperature accuracy and stability below 4.2 K helium boiling point 	 Loses temperature control when transitioning through 4.2 K helium boiling point Control of temperature at (and near) 4.2 K helium boiling point is relatively less stable than with CTLC mode

Table 3-1. Characteristics of low-temperature control modes

³ CLTC cooling problems are most often from frozen contaminants blocking one of the impedance tubes. To clear the tube, you must remove the entire PPMS probe from the liquid helium dewar.

3.3.2 Temperature-Approach Modes

You can set the PPMS to approach a temperature set point with the **Fast Settle** mode or the **No-Overshoot** mode. **Fast Settle** mode changes the temperature very rapidly, but it can undesirably affect samples that show temperature hysteresis behavior. In **Fast Settle** mode, the temperature-control hardware first overshoots or under-shoots the temperature set point in order to help overcome thermal gradients in the sample chamber, then the hardware backtracks to the set point. In **No-Overshoot** mode, the PPMS approaches the temperature set point slowly, from only one direction, so it does not overshoot or undershoot the set point. Thermal equilibrium takes considerably longer to achieve in **No-Overshoot** mode.

3.4 Atmospheric Control

The PPMS vacuum and gas lines accommodate thermal control and atmospheric control of the sample chamber. Figure 3-3 illustrates the vacuum and gas lines.



Figure 3-3. Gas and vacuum control in the PPMS

The flow-control valve and the differential pressure sensor are part of the PPMS temperaturecontrol system. The flow-control valve, which is between the pump and cooling annulus, adjusts the rate at which helium vapor is drawn across the sample chamber. The differential pressure sensor, which is between the annulus and dewar, monitors the pressure difference between the annulus and dewar in order to facilitate filling the annulus with helium for low-temperature operation. All other system plumbing is used for atmospheric control. Under normal operation, the flush valve opens the sample chamber to the pump. The vent valve allows helium gas into the sample chamber during venting and purging procedures. A purging procedure vents the sample chamber with clean gas and pumps it out through the flush valve, and then repeats the same process two more times. When the chamber is sealed, both the vent valve and flush valve are closed. The vent and flush valves are never open at the same time. The system's solid-state silicon pressure sensor is located on the sample space line within the Model 6000, between the vent valve and flush valve, as shown in Figure 3-3.

Gas-Line Configuration

The standard PPMS configuration does not provide a high or ultra-high vacuum environment. It keeps the sample chamber at a few torr with gaseous helium vapor supplied by the dewar. However, the gas lines can be configured in several different ways. For example, you can substitute some other clean gas for the helium that vents the sample chamber and provides the several torr of pressure that maintains thermal uniformity within the sample space. The alternate gas source should be connected to the "Sample Vent-Up" port that is on the rear of the Model 6000. When you use an alternate gas source, you should blank off the "Gas Source" port, which provides helium from the dewar, so that the differential pressure sensor will still work. Additional plumbing and gauges can also be inserted between the pump and flush valve.

If a high or ultra-high vacuum is required in the sample space, you can insert an alternate type of vacuum pump into the system near the probe head, where larger throughputs can be achieved. The efficiency of sample-space temperature control can be adversely affected by changing the pressure of gas within the sample chamber. However, rather than thermal control through heat exchange, some experiments require an adiabatic environment (e.g., heat capacity). Quantum Design offers a High-Vacuum option for the PPMS for precisely such requirements.

3.5 Magnetic-Field Control



WARNING!

The helium level must be **above** the superconducting magnet (a helium level of about 60%) to take the magnet to full field. There is high potential for damage, such as an uncontrolled magnet quench, when the superconducting magnet is not completely covered by helium. See Sections 1.4.2 and 4.2.4 for more information.

Figure 3-4 illustrates how the current through the magnet coil is changed to charge or discharge the magnet. The essential process is as follows:

- 1. The Model 6700 Magnet or Model 3120 Controller matches the current in the magnet.
- 2. A small portion of the superconducting magnet wire (the persistence switch) is heated by another resistive wire.
- 3. The heated persistence switch becomes non-superconducting, which switches the magnet controller into the previously closed superconducting circuit.
- 4. The magnet controller drives the magnet to the current that is necessary for the new field.



Figure 3-4. Changing the field in the magnet: To change the field in the magnet from H_0 (a) to H_f (f), the magnet power supply first matches the magnet current (b). Then the persistent switch heater is turned on (c), switching the power supply into the circuit. The current is driven to the new value (d) and the persistent switch heater is turned off (e).

The persistence switch heater is generally turned off after the field set point is reached, allowing the entire magnet to superconduct again. The magnet is in **Persistent** mode when the persistence switch is superconducting. In **Persistent** mode, the current in the magnet does not dissipate, so the power supply current can be turned off. The magnet can also be operated in **Driven** mode, which retains the current source in the magnet circuit in order to drive the current. Field changes can be made more quickly in **Driven** mode, but the resulting field is much noisier.

3.5.1 Control Mechanisms

For each PPMS, the field in the sample space is a known function of the current in the magnet. Systems are individually calibrated to their own field-to-current ratio. To ensure that the proper field exists in the magnet during magnet charging and discharging, the current from the power supply is passed through one of two calibrated resistors magnet controller before the persistence switch heater is turned off. The two resistors are for high-power and low-power operation, and the appropriate resistor is used for each current. The voltage drop across the resistor is directly proportional to the current in the magnet and thus proportional to the field within the sample space. The field is calculated from this potential drop and if the field is not within a certain range of the field set point, the magnet current is adjusted accordingly until the field is within the acceptable range. For 7-T and 9-T longitudinal magnets, the field must be within about 1.5 Oe of the set point for set points above about 9500 Oe. For set points below 9500 Oe, the field must be within about 0.15 Oe of the set point before the persistence switch heater is turned off. For 14-T magnets, the field must be within about 3 Oe of the set point for set points above about 15,000 Oe, and the field must be within about .3 Oe of the set point for set points below 15,000 Oe before the persistence switch heater is turned off. The field that is reported is calculated from the drop across the resistor. The temperature coefficient of the calibration resistors in the Model 6700 is nominally 30 ppm/°C, so variations in the temperature of the instrument might have very small effects on the reported field. Notice that the field reported by the PPMS is only that due to the current through the magnet circuit—the reported field value does not account for any background sources or remnant field in the magnet.

After the persistence switch heater is turned off, magnetic field relaxation, or flux creep, can still occur. To minimize this effect, the PPMS offers a specific magnet-charging technique called **Oscillate** mode, which is described in Section 3.5.

3.5.2 Magnetic-Field Approach Modes

The PPMS uses **Oscillate** mode, **No-Overshoot** mode, or **Linear** mode to approach a field set point.

In **Oscillate** mode, the magnet controller allows the magnet to overshoot or undershoot the field set point by about 30%, if possible, and then narrows in on the set point in an oscillatory fashion, under-shooting or overshooting the set point by 30% on each iteration. **Oscillate** mode can undesirably affect samples that show field hysteresis behavior. **Oscillate** mode is best used to help eliminate field relaxation. When charging to zero field, the **Oscillate** mode should be used to keep the remnant field in the magnet as small as possible.

In **No-Overshoot** mode, the magnet is charged to 70% of the difference between the field set point and the present field, and then the magnet slowly approaches the set point, from only one direction, in continuous 70% increments until the magnet is close enough to the set point to drive directly to it without overshooting it. The charging direction is never reversed when **No-Overshoot** mode is used, but field relaxation can occur. That is, after the magnet enters **Persistent** mode, the actual field in the magnet can change slightly from the reported field. You should use **No-Overshoot** mode with field-hysteretic samples.

Linear mode is the quickest charging mode. **Linear** mode fine-tunes the field after an initial attempt at charging the magnet directly to the field set point. Both field overshooting and field relaxation are possible in **Linear** mode.

Important: When you set measurement parameters, do not confuse the **No-Overshoot** temperature-approach mode with the **No-Overshoot** magnet-charging approach mode.

3.6 Helium-Level Metering

The helium-level meter is inside one of the rods running the length of the probe. The meter is thus outside the sample chamber and vacuum tubes. The helium-level meter is a long superconducting wire configured for a four-wire resistance measurement. Because the portion of superconductor that is not immersed in liquid helium is resistive, the resistance of the wire is directly proportional to the amount of liquid helium required to fill the tank. The value that the PPMS reports is a percentage of full. For example, 100% indicates the dewar is full, 75% indicates the dewar is three-quarters full, and so on. The helium-level meter does not extend all the way to the bottom of the dewar, so 0% does not mean the dewar is dry, only that the meter is completely exposed. When the helium-level meter is completely exposed, the impedance tube intake of the probe is not immersed in liquid helium, and temperature control will be lost or inhibited.

Heat is generated by the metering process, so the helium-level meter is usually not on continuously, but the helium level is automatically checked on an hourly basis. You should monitor the helium continuously only during helium transfers. For more information about continuous monitoring, refer to the "Helium Level" section in the *Physical Property Measurement System Commands Manual.*



 $LEVEL = 100\% - R/R_{0}$

Figure 3-5. Schematic of helium-level meter

3.7 Model 6000 Flexibility

The electrical inputs and outputs on the Model 6000 allow the PPMS to accommodate a wide range of experiments and to be configured to fit many different needs. For example, you can monitor the channels of the user bridge from the Model 6000 analog outputs or front panel or from the personal computer. Appendix A lists all Model 6000 inputs and outputs and explains the uses and capabilities of each port so that you can customize the system.

The voltages of the four Model 6000 analog outputs can be linked to 30 different sources with desired gain settings. These analog outputs allow use of an oscilloscope, chart recorder, or similar instrument. The "Link BNC to Parameter" section in the *Physical Property Measurement System Commands Manual* explains how you configure the analog outputs.

The Model 6000 provides three -24 V auxiliary drives (relays); a low-current, 15 V power source; two different types of digital TTL level inputs; two -10 V to +10 V analog inputs the Model 6000 can digitize; and three optically isolated 5 V external select lines (TTL-level outputs). Model 6000 units that have a user bridge board also have access to two current drivers that provide up to 1 A or 20 W of power—whichever limit is reached first. You can configure each input and output.

The Model 6000 also provides two communication ports (IEEE-488 and RS-232); a motor output that includes connections for a 0/-24 V actuator, index, and limit switch; and a configurable pressure gauge input. Besides electrically configuring the system as necessary, you can also configure the gas and vacuum lines as required, adding gas sources, gauges and pumps (see Section 3.4). The complexity of the system allows several different ways to set up the same experiment.

The automated sequence feature of the PPMS lets you automate the entire measurement process. Any function the Model 6000 can perform, including controlling all PPMS hardware and recording measurement values, can be accessed within a sequence file that runs automatically, so you can perform an experiment without being present. Try exploring this feature. The "Sequence Files" section in the *Physical Property Measurement System Commands Manual* discusses measurement automation in detail. The *Physical Property Measurement System: PPMS MultiVu Application User's Manual* discusses how to use PPMS MultiVu sequence files and sequence commands.

If the Model 6000 does not have the specifications necessary for your experiment, you can still measure a sample in the PPMS by using your own instruments. Appendix A lists the pinouts for the sample puck, probe head Lemo connectors, and Model 6000 "D" connector so that you can make the necessary electrical connections. You can attach a current source and voltmeter to the sample to perform four-wire resistivity measurements. By attaching the sample leads to the sample in a slightly different configuration, you can make Hall coefficient measurements. You can use other instruments to measure other sample characteristics. You can even interface GPIB-capable instruments with the Model 6000 and a PC in order to facilitate automated data collection from *other* instruments. If you have questions about how to customize the PPMS to fit your specific application, contact a Quantum Design representative. Quantum Design offers a variety of options designed specifically to help meet the needs of your experiments.

3.8 Example Measurement

The PPMS can be configured for four-terminal resistance, Hall effect, magneto-resistance, critical current, critical field, critical temperature, DC magnetization, AC susceptibility, heat capacity, and thermal conductivity measurements—to name some of its more common uses. Quantum Design is continually developing PPMS options that standardize frequently made measurements, making them easier to perform and more accurate. You use each option in conjunction with the PPMS in a different manner.

One of the most common measurements made with the PPMS is resistivity. Quantum Design offers more than one resistivity option for the PPMS. This section describes a resistance measurement to illustrate how you can use the PPMS.

To perform four-terminal resistance measurements, you mount a sample on a puck and attach four leads to the sample. An example is shown in Figure 3-6; you can use other geometrical arrangements of the leads.



Figure 3-6. Leads attached for four-terminal resistance measurement

Solder each lead to an appropriate solder pad on the base of the puck. This allows the hardware to make electrical contact with the sample. In the Model P400 Resistivity option, the hardware that makes electrical contact with the sample is the user bridge board in the Model 6000, and the solder pads you use are #3, #4, #5, and #6 (see Appendix A). To perform simultaneous measurements on another sample, the other sample would be mounted to the same puck and its leads would be soldered to pads #7, #8, #9, and #10. Take care that you electrically isolate each sample and each sample lead. The puck is conductive, so the leads must be insulated and samples must be mounted with an electric, but not thermal, insulator underneath them. Most PPMS options also allow you to use special pucks that have labeled solder pads prewired to the surface of the puck for easier sample mounting. Some options, such as the ACMS option or the Horizontal or Vertical Rotator option, use different sample holders.

You will insert the puck into the sample chamber by using the procedures explained in Section 4.5. During the measurement the hardware will pass a current through the sample via two leads, using the other two leads to measure the electric potential drop across the sample. Because the input impedance of the voltmeter is very high, both the current and the potential drop can be known to a high degree of accuracy. Ohm's law gives the resistance. The user bridge board operates in several different modes to accommodate a variety of requirements and still allow a

high degree of measurement sensitivity. The resistance that the bridge board measures is reported in the **Status–Bridge** screen on the front panel of the Model 6000.

To perform different types of measurements, different hardware is used, but the basic premise of the system remains the same. The sample sits within the thermally, magnetically controlled environment of the sample space while electrical wiring to the base of the chamber allows connection to current sources, voltmeters, and the like. The Model 6000 frequently contains all such necessary equipment.

You can manipulate various measurement parameters, including the measured resistance, applied current, sample temperature, applied magnetic field, time, select line status, and so on. These values can be placed into a data buffer in the Model 6000, linked to one of the Model 6000 analog outputs, or uploaded into a data file on a personal computer. The PPMS software can graph the data file as the data is recorded. You can export data files to another format in order to use them with data manipulation applications, such as spreadsheets or professional graphing programs. If you are using the PPMS as a temperature and field control platform, you can measure sample resistance as a function of temperature and magnetic field. Entire resistance curves can be plotted and the critical temperature can be determined for superconducting samples.

For the example given above, you could set the PPMS to measure the sample resistance at 20 different temperatures and then increase the applied magnetic field by 0.25 T and repeat the resistance measurements at all 20 temperatures. This type of measurement can be facilitated by using sequences.

When you use a sequence file, the PPMS becomes fully automated. It can automatically perform temperature changes, field changes, applied current changes, and resistance measurements. It can automatically control all PPMS hardware and thus place the system in **Shutdown** mode when the experiment is complete. The Model 6000 can control numerous other operations. Refer to the *Physical Property Measurement System Commands Manual* and the *Physical Property Measurement System Commands Manual* for further information regarding sequence commands. The main point of illustration here is that the above resistance experiment can be performed entirely automatically. You can automate other experiments in a similar manner.

3.9 Experimental Considerations

Although the PPMS is extremely flexible, you must consider certain limitations when you design an experiment. One of the first constraints to consider is the size of the sample. The diameter of the sample puck is 2.3 cm, with a set of notches around its perimeter in which the electrical leads seat. The maximum sample height is 5.0 cm. While the superconducting PPMS magnets have a high field homogeneity, the uniformity of the field over the sample is greater for small samples. Similarly, although the PPMS provides very precise temperature control, the effect of thermal gradients on the sample is less for small samples. Recall that temperature control in the PPMS is usually based on the temperature of the sample puck. Settling times, before the sample is at the same temperature as the puck, also could be longer for large samples

The PPMS is ideally suited for measurements of bulk solid samples and thin film samples. Powdered, aqueous, and liquid samples can be accommodated with a variety of techniques, but you should use caution with such techniques, because the PPMS is very difficult to clean in the event such a sample is lost within it. The use of sealed sample holders requires additional caution, because they tend to burst when the sample chamber is evacuated. Before introducing a sealed sample holder into the PPMS, you should verify that it will not break when it is subjected to an external pressure of only a few torr, as it will be inside the PPMS sample chamber. When you mount a sample, it is important to secure it with a method that will withstand the experimental extremes. Be sure to determine the thermal, magnetic, and conductive properties of the bonding media before using it in an important experiment. The temperature range of the PPMS is 1.9–400 K. The magnetic field available depends on the magnet that is purchased with the system. Other properties of the bonding media and of any electrical leads could also be important. For example, with thin film samples, the leads and bonding media must not chemically react with the sample.

The sample puck is conductive, so when you use it as a mounting technique, verify that the electrical leads are isolated and individually insulated. When using the sample puck you also need to consider how the sample might interact with it—often, samples must not be in electrical contact with the puck. Note that thermal contact with the puck is still desired in the latter cases. You can use a substance such as sapphire to electrically isolate resistive samples from the puck and still allow good thermal contact. A thin layer of Kapton tape serves as a less expensive substitute. For experiments that use other sample holders, you should consider the relevant properties of the sample holder. For example, for DC magnetization measurements, you can use a clear plastic drinking straw as a sample holder because the straw fits within the sample chamber, is easy to use with option hardware, is not conductive, and has very low magnetic susceptibility.

System Operation

4.1 Introduction

This chapter contains the following information:

- Section 4.2 presents general guidelines for using the PPMS.
- Section 4.3 describes how to change the low-temperature control mode (pot fill or CLTC).
- Section 4.4 discusses sample-mounting procedures.
- Section 4.5 explains how to install a puck into the sample chamber and how to remove it.
- Section 4.6 describes some of the ways the PPMS can be customized.
- Section 4.7 describes routine maintenance procedures such as refilling a cold dewar and checking the oil in the vacuum pump.

4.2 General Guidelines

The PPMS is a precision laboratory instrument that is designed to be robust and adaptable. However, it is complex, and some parts are fragile. This section provides guidelines for the appropriate use and maintenance of the system and its critical components. You can help prevent damage to the system and ensure that it provides optimal measurements by reviewing this material and following the guidelines.

0

4.2.1 Handling the Probe



WARNING!

Always remove the probe from the dewar very slowly—raise it about one inch per minute. The probe could explode violently if you rapidly pull it from the dewar when there is a leak in the vacuum space.

Handle the probe with care; it is an intricate, delicate, and expensive piece of equipment. Always use the plate just below the probe head (Figure 2-4) to support the probe. The long tubes that run between the probe head and the magnet end of the probe are part of the equipment—they are *not* structural supports, they *cannot* support the full weight of the probe, and they are easily damaged. Always provide support at both the magnet end and the probe head when you lay the probe in a horizontal position.

It is important to work slowly and carefully when you lower a probe into (or lift a probe out of) a full or partially full dewar. To facilitate this process, Quantum Design includes a probe-lifting assembly with the system (see Section 4.7.4). By gradually lowering the probe into the dewar, you decrease the unnecessary helium boil-off caused by a warm probe. It also can avoid serious damage to the equipment that could occur if a part of the probe froze or boiled because of unexpected circumstances, such as a leak. In addition, you can watch for unusual behavior, such as condensation or gas escaping from relief valves, when you slowly move a probe in and out of liquid helium.

Keep the original packing crate and padding for the probe so that you can use it in the event you ship the probe back to Quantum Design for modification, option installation, or repair.

4.2.2 Powering the System Off and On

Generally, the power to the PPMS hardware should be left on—including the power to the Model 6000, Model 6700, and vacuum pump—to maintain system safeguards, as explained in Section 4.2.2.1. In the event you must turn off the power to the system or any component, use the sequence in Section 4.2.2.2. Turn on the system or component as soon as possible, using the sequence in Section 4.2.2.3.

Important: Before you turn off the power to the Model 6000, verify that the system is in **Shutdown** mode with the magnet in **Persistent** mode and the **Field** at zero (0) Oe. Also, leave the magnet leads and blue Lemo attached to the system.

If there is an *unplanned* power outage, we recommend that you turn off the power to all components and the main system breaker, in that sequence, then pull the power plug from the wall. Leave the magnet leads and blue Lemo attached to the system. When the power returns, turn on the system by using the sequence in Section 4.2.2.3.

You do not need to turn off the system if it will be idle, but you can conserve helium by putting it in **Shutdown** (standby) mode (Section 4.2.3).

Important: Shutdown mode *does not* turn off the system, it reduces the use of helium while allowing the Model 6000 to monitor the status of the system.

4.2.2.1 POWER LOSS

During a power loss or when you power off the system, leave the magnet leads connected—the leads will allow any current in the magnet to safely drain away. Also, leave the blue Lemo connected to the system.

Important: Do not disconnect the blue Lemo or the magnet leads while the power is off.

When the power to the Model 6000 is cycled off and back on, the Model 6000 retains certain parameters, including all information stored in the sequence file and data buffer, the field in the magnet, and most of the user-configuration parameters. To support the nonvolatile RAM, the Model 6000 has a lithium battery that lasts about 10 years.

When there is a power loss, the software will automatically place the PPMS in **Shutdown** (standby) mode, but the PPMS will lose the settings for any measurements (e.g., temperature, field) that are in process at the time of the outage. Further, the system will stop sending output to any motor. Other types of information that will not be retained or restored include commands that are being executed when the power is lost as well as any direct output from the Model 6000, such as analog outputs, which return to 0 V. If a power outage occurs when you are using a sequence to perform measurements, the sequence will stop running. When the power comes back on, you will need to reset the measurement parameters and restart the measurement.

When the power to one or more components is turned off, the system cannot effectively monitor its own status. For example, the Model 6000 assumes that all other components are present and functioning, even if a component has been powered off. If you turn off the power to the Model 6000 for several hours while the flow-control valve is open, the pump will stay on, filling the cooling annulus with liquid helium. As a result, the system will require an unusually long time to warm to above 4.2 K when you turn on the Model 6000. We recommend that you use the sequence below to turn off the PPMS, and that you always place the PPMS in **Shutdown** mode before you turn it off—these procedures will help bring the system to a stable, helium-conserving state.

4.2.2.2 POWER OFF SEQUENCE

- 1. If the magnetic field is not in **Persistent** mode and at zero (0) Oe, reset it according to the sequence below:
 - a. Select **Instrument >> Field** (Figure 4-1).
 - b. In the **Field** dialog box, set the **Mode** to **Persistent** and the **Set Point** to zero (0) Oe.
 - c. Click on the **Set** button.
 - d. Leave the dialog box open so that you can monitor the field until it is within 1000 Oe of zero (do not continue until the field is within 1000 Oe of zero).
 - e. In the **Field** dialog box, click on the **Close** button.
- 2. Bring the system to a stable state by putting it in **Shutdown** mode:
 - To use the Model 6000, select CTRL >> Interactive Control >> 8. Shutdown Mode.
 - To use MultiVu, select **Instrument** >> **Shutdown** from the dropdown menus at the top of the MultiVu window.
- 3. Deactivate any active option (Utilities >> Activate Option).
- 4. Exit the MultiVu program and turn off the power to the computer.
- 5. Disconnect the annulus line (the large pumping line) at the probe head. Open the annulus connection enough to stop the flow, but leave it seated in the connector.
- 6. Turn off the power to the individual PPMS components, including the vacuum pump.
- 7. Turn off the main breaker on the back of the PPMS cabinet.
- 8. Unplug the PPMS plug from the power source.



Figure 4-1. PPMS Field dialog

4.2.2.3 POWER ON SEQUENCE

- 1. Plug in the PPMS power cord.
- 2. Turn on the main breaker on the back of the PPMS cabinet.
- 3. Turn on the power to the computer.
- 4. Turn on the power to the individual PPMS components, including the vacuum pump.
- 5. Start MultiVu.
- 6. Set the temperature to 5 K (**Instrument** >> **Temperature**). When you set the temperature to 5 K, the equipment automatically opens the valves and starts pumping out the lines.
- 7. Wait five minutes for the system to stabilize.
- 8. Reconnect the annulus line at the probe head.
- 9. Set a new temperature.
- 10. Activate an option.

4.2.3 Shutdown Mode

Shutdown (also known as standby) mode *does not* turn off the system, but it does help conserve helium resources while allowing the Model 6000 to monitor the status of the system. Place the PPMS in **Shutdown** mode whenever it will be idle and you want to conserve helium.

When the temperature-control hardware is in **Shutdown** mode, the software adjusts the flowcontrol valve to maintain approximately 100 cc/min. of flow through the cooling annulus, turns off the system heaters, and lowers the power to the impedance heater. Note that with these adjustments, the PPMS does not remain at a steady temperature.

Important: The magnet must be in **Persistent** mode and the **Field** must be at zero (0) Oe *before* you place the system in **Shutdown** mode (see Step 1 in Section 4.2.2.2).

When you initiate the **Shutdown** mode, the software automatically places temperature control in standby mode and seals the sample chamber. These settings will be displayed in the front panel of the Model 6000 and in the **Status** bar at the bottom of the MultiVu window.

- To put the system in Shutdown mode using the Model 6000, select CTRL >> Interactive Control >> 8. Shutdown Mode.
- To put the system in **Shutdown** mode using MultiVu, select **Instrument** >> **Shutdown** from the dropdown menus at the top of the MultiVu window.

To end **Shutdown** mode, set a new temperature (in MultiVu, select **Instrument** >> **Temperature**).

4.2.4 Monitoring the Helium Level

The helium level in the dewar must be regularly monitored, especially if you are using magnets—*the helium level must be above about 60%* to charge magnets to high fields. If you do not have or are not using a magnet, you can let the helium level drop to approximately 30% before refilling the dewar, as explained below. Section 4.7.2.2 has instructions for refilling a cold PPMS dewar with liquid helium.

In many cases, you can safely transfer helium into the PPMS dewar while a sequence is running. *Do not* add helium to the dewar if you are ramping the magnet or if the temperature is below 5 K.

4.2.4.1 HELIUM LEVELS: USING A MAGNET



Figure 4-2. Helium levels relative to probe and 9-T magnet

The PPMS is not like a car—although you can drive a car until the fuel gauge reads nearly empty, the

PPMS could be seriously damaged if you operate the magnet when it is not immersed in liquid helium: When a charged superconducting magnet is not completely immersed in liquid helium, there could be an uncontrolled magnet quench, warming the magnet so that it loses its superconducting properties and gives off large amounts of energy in the form of resistive heat.

Figure 4-2 shows approximate helium levels relative to a 9-T PPMS magnet (the exact location of the top of the magnet varies from magnet to magnet). *To ensure that the magnet remains immersed, you should perform a helium transfer whenever the helium-level meter reads below about 60%.* As explained in the next section, the helium level does not change at a consistent rate. In the event you plan to let the helium level drop below 60%, verify that the magnet does not have a persistent field.



WARNING!

Keep the helium level **above** the superconducting magnet (a helium level of 60%). There is high potential for damage, such as an uncontrolled magnet quench, when the superconducting magnet is not completely covered by helium. See Sections 1.4.2 and 3.5 for further information.

4.2.4.2 HELIUM LEVELS: NOT USING A MAGNET

When you are not using a magnet during measurements, we recommend that you begin carefully monitoring the helium level when it reaches 30%. Maintaining the helium level at 30% or above will help prevent serious temperature-control problems (see below). Although typical static helium boil-off rates are usually less than 5–7 liters per day, the actual rate of PPMS helium consumption varies, depending on ambient conditions and how the system is being used. For example, when the helium level reaches 30%, it begins decreasing faster than when it is above 30%—the shape of the dewar interior and the nature of the helium-level meter mean that the absolute boil-off rate does not translate into a constant percentage drop in the helium-level meter. Levels appear to drop faster when the dewar is full or almost empty because the helium container is narrower at the top and bottom.

Carefully consider helium-consumption rates before you start a long experiment, especially when the PPMS will be running an unsupervised automated sequence. If you are not using a magnet during measurements, you can maintain temperature control with helium levels as low as 12%. Below about 12%, the helium bath no longer covers the impedance tube and the system quickly loses the ability to control temperature. Further, contaminants can enter the impedance and create a blockage, which will cause additional problems. Because changes are so unpredictable by the time the helium level reaches 12%, it is difficult to accurately monitor low helium levels. For these reasons, we recommend maintaining the helium level around 30% at all times, except for prolonged idle periods.

4.2.5 Monitoring the Nitrogen Level

The nitrogen in nitrogen-jacketed dewars serves a less crucial purpose than the helium, so it could boil away almost completely without any repercussions other than more rapid consumption of helium. However, to keep the helium well insulated, you should fill nitrogen jackets about twice a week. Section 4.7.2.1 explains how to transfer liquid nitrogen into a cold PPMS dewar.

To determine if nitrogen remains in the jacket, look for ice on one of the pressure-relief valves. You can check the nitrogen level by dipping a clean, frosted, metal rod into the jacket through one of the nitrogen fill ports. To frost the rod, dip it in liquid nitrogen, then expose it to room-temperature air.

4.3 Setting the Low-Temperature Control Mode

The Quantum Design PPMS offers two unique modes for controlling low temperatures in the sample chamber: Continuous Low-Temperature Control (CLTC) and pot-fill (these modes are explained in Chapter 3). Since January 1998, the PPMS has included both modes, with CLTC shipped as the default. Earlier model PPMS systems included only the pot-fill mode, but owners of such systems can purchase the CLTC option.

If your PPMS has the capability for both low-temperature control modes, you can change from CLTC to pot-fill mode (or vice versa) by using the MultiVu **Utilities** dropdown menu or the Mon6000 utility, which is often located in C:\QDPPMS\Tools. Here we include instructions for using both utilities. To determine if you have the CLTC option, follow the MultiVu instructions in Section 4.3.1.1 through Step 3.

4.3.1 MultiVu

As explained below, you will first verify the low-temperature control mode that is currently active, then you will issue the command to switch to the other mode.

4.3.1.1 VERIFY OPTION AND CONTROL MODE

In MultiVu, you will use the **Send GPIB Commands** dialog to verify the mode that is being used.

 Select Utilities >> Send GPIB Commands from the MultiVu dropdown menus (Figure 4-3).



Figure 4-3. Opening the Send GPIB Command utility in MultiVu

2. The Send GPIB Commands dialog will open, as shown in Figure 4-4.

Send GPIB (Commands		×
Send:	clt?	Device:	15
Command Terminator:	● None		
Response:	0.1.1		4
Result	No error.	Bytes:	4
	Send Send and Read Close		

Figure 4-4. Using the Send GPIB Command dialog to verify control mode

In the **Send GPIB Commands** dialog, note the **Send**: text box at the top of the dialog, the **Response**: area in the middle of the dialog, and the **Send and Read** button at the bottom of the dialog. You will use these sections to verify the temperature-control mode.

3. In the text box next to **Send:** type the following: clt? (just as is shown in Figure 4-4). Then, click on the **Send and Read** button. In the **Response:** area, the utility will report two numbers (e.g., "0,1" as shown in Figure 4-4).

The first number indicates the active temperature-control mode and the second indicates if the CLTC option has been installed. For example, the first number in Figure 4-4 is "**0**," indicating that the system is using pot-fill mode, and the second number is "**1**," indicating that the CLTC option has been installed. A "**0**,**0**" report would indicate that pot-mode is being used but there is no CLTC option, and a "**1**,**1**" report would indicate that low temperatures are being controlled by the CLTC option. The status codes are summarized in Table 4-1.

Table 4-1. Status codes for temperature-control modes

STATUS CODE	TEMPERATURE- CONTROL MODE	CLTC INSTALLED	
0,0	Pot fill	No	
0,1	Pot fill	Yes	
1,1	CLTC	Yes	

4.3.1.2 CHANGE LOW-TEMPERATURE CONTROL MODE

To use MultiVu to change the low-temperature control mode, you will issue a command to shut down the PPMS, along with a number specific to the control mode that you want to be activated. The shutdown commands are summarized in Table 4-2.

Sand GPIR Commands

The example below uses the commands that activate CLTC.

1. In the **Send:** text box type the following:

shutdown 2

(just as is shown in Figure 4-5).

2. Click on the **Send and Read** button.

Send:	shutdown	2			•	Devi
Command Terminator:	None	C CR/LF	O LF	C Custom	;	
Response:						
Result:						Bytes:
		Send	Send and I		Close	-

Figure 4-5. Switching from pot-fill mode to CLTC

- 3. To verify which temperature mode the system has activated, type clt? and click on the **Send and Read** button again (Figure 4-6).
- 4. If the system has switched to CLTC, the **Response** area should now display "**1**,**1**" (Figure 4-6).

Send GPIB (Commands						×
Send:	clt?				•	Device	e: 15
Command Terminator:	None	C CR/LF	O LF	C Custom	;		
Response:	1.1:						Ā
Result:	No error.					Bytes:	4
	2	Bend	Send and I	Read	Close		

Figure 4-6. Verifying that CLTC is the low-temperature control mode

COMMAND	COMMAND ACTION	
shutdown 0	Shut down the PPMS but do not change the temperature-control mode.	
shutdown 1 Shut down the PPMS and set the temperature-control mode to pot fill.		
shutdown 2	Shut down the PPMS and set the temperature-control mode to CLTC.	

Table 4-2. Commands to shut down the PPMS and set the temperature-control modes

4.3.2 Mon6000

The **Mon6000** dialog is set up somewhat differently than the **Send GPIB Commands** dialog in MultiVu, but you will use the same commands that you use with MultiVu (Table 4-1) to verify or change the low-temperature control mode. Note that you need to press the **<Enter>** key after you enter each command into the **Mon6000** dialog.

- 1. Open the **Mon6000** dialog:
 - a. Locate the Tools subdirectory of the QDPPMS directory (the QDPPMS directory might be on your C: drive).
 - b. Locate the file named Mon6000.exe and double click on it to open it.
- 2. When the Mon6000 dialog opens (Figure 4-7), you will see separate text-entry panels titled "Command To Send" and "Response Received."

🚥 Mon6000 for Win32		
<u>File A</u> cquisition <u>S</u> earch Abo	put	
Command To Send Auto-	1 sec <u>Response Received</u>	
clt?	1.1;	

Figure 4-7. Checking the low-temperature control mode using the Mon6000 dialog

3. Enter your temperature-mode verification command (i.e., clt?) into the **Command To Send** panel and press the **<Enter>** key.

As shown in Figure 4-7, the **Response Received** panel will then display the active lowtemperature regime. You can see that the CLTC option is installed and it is being used, because the **Response Received** panel displays "1,1" just as it would in MultiVu.

4. To shut down the PPMS and activate pot-fill mode, enter the shutdown command (shutdown 1), as is shown in Figure 4-8, and press the **<Enter>** key.

🚾 Mon6000 for Win32		. 🗆 ×
File Acquisition Search About		
Command To Send Auto-1 sec	Response Received	
shutdown 1		

Figure 4-8. Switching low-temperature control modes using the Mon6000 dialog

5. You can verify that you have changed to pot-fill mode by typing "clt?" again in the **Command to Send** panel.

The **Response Received** panel should display "**0,1**" if the system has changed to pot-fill mode successfully.

6. To activate CLTC, type shutdown 2 in the Command to Send panel.

4.4 Sample Mounting

Several broad considerations affect the sample-mounting technique you will use, no matter what type of sample holder you choose, including a sample puck and a plastic straw. These considerations are the temperature range of the experiment and the electrical and magnetic properties of the elements in the mounting arrangement.

4.4.1 Guidelines for Mounting Samples

The sample-mounting method that you use must withstand the temperature range of the experiment. Not all glues and tapes stick well at low temperatures. Furthermore, differential thermal expansion between elements of your arrangement could prevent it from functioning as you intended.

The sample puck is conductive, so electrical leads will be shorted together if they contact the puck anywhere other than at the solder pads. Often, samples must be electrically isolated from the puck so that the only electrical path is through the sample. Other sample holders, such as the PPMS rotators, are made from dielectric material and do not short the signals when a sample contacts them directly. If the PPMS will be used as a magnetometer, knowing the magnetic moment of the sample holder is also important. To determine its effect on the sample measurement, measure the magnetic moment of the sample holder without a sample.

In general, samples must be securely mounted to the sample holder so that their position will remain constant (or known, in some cases). Secure mounting also is important to help prevent losing samples inside the sample chamber—it usually requires great effort to retrieve lost samples and to clean the chamber.

4.4.2 Mounting a Sample on a Sample Puck

There are many methods to secure a sample to a puck. Leads are frequently soldered or welded to samples, but the type of wires used and the method of contact vary by application. In addition, tapes, conductive epoxies, greases, glues, and paints can be used. Conductive pads can be coated onto semiconducting, thin film samples. Each method can have an array of thermal, electrical, magnetic, and reactive properties.

If you will be using a puck as the sample holder, first consult Appendix A or the appropriate option manual to determine the proper solder pads to use for electrical contact. Also, plan the geometrical arrangement of the leads.

It will be easier to solder leads to the solder pads if you remove the connector PC board from the bottom of the puck. To do so, remove the screw from the bottom of the puck. The base of the puck is slotted so that the two components fit together properly when reattached.



Figure 4-9. Sample mounted on puck



Figure 4-10. Intermediate sample leads

Verify that the leads all pass through the notches on the edge of the puck, as shown in Figure 4-9. Note that if the leads extend past the outer rim of the puck, it will be easy to damage them with the puck-insertion tool during puck insertion. Also, you should use insulated electrical leads because the puck is conductive.

In some cases it can prove useful to create an intermediate set of leads that contact the solder pads but stop short of the sample, as illustrated in Figure 4-10. You can then treat the sample and another set of leads—for example, in an oven or coating chamber—while isolating the puck from the treatment. When you have completed preparation of the sample and leads, you can secure them to the puck and attach the sample leads to the intermediate leads.

To prevent electrical signals from being shorted, you must separate the sample and the conductive puck by a substance that has high resistivity compared to the sample. In many cases, this substance can be a piece of tape. Take the phase characteristics of the insulating material into consideration, because the material will not perform correctly if it conducts or superconducts in the temperature range for your experiment. The thermal conductivity of an insulator also might be important, depending on the nature of the experiment.

It is often important that the sample and puck are in good thermal contact, which ensures that the temperature of the system thermometer accurately represents the temperature of the sample. If the sample is in poor thermal contact with the puck, heat conduction will occur through the leads and through helium gas, which is significantly slower than through the puck.

The surface of the puck can be machined to the desired geometric characteristics. To do this, you first remove the screw from the bottom of the puck and take off the connector PC board so that it will not be damaged during machine work. Work carefully so that you do not alter the edge of the puck, where the key and the groove for the puck-insertion tool are located. Also, you can remove the connector PC board from the bottom of the puck before you perform other puck treatments, such as heat treatment.

If you want to verify proper electrical connection of the sample before you insert the puck into the sample chamber, you can use a digital voltmeter or similar instrument after you have mounted the sample. Using Figure 4-11 as an example, gently contact the gold-plated receptacles on the bottom of the puck with the meter probes.



Figure 4-11. Checking for proper electrical connection of the sample

You can use this technique to check for undesired shorts, poor connections, and so on, or you can insert the puck into a P150 sample-wiring test station¹ and use the numbered banana jacks for the same purpose. Notice that the puck plugs into a plastic ring in the test station, so any shorts to ground can only be measured by directly contacting the puck itself. However, when the puck is plugged into the sample chamber, it contacts metal and is truly grounded. To ensure the validity of your data, verify that there are no shorts to the surface of the puck.

4.5 Sample Puck Installation and Removal

4.5.1 Installing a Sample Puck

After you have mounted the sample on the puck and soldered the leads to the appropriate solder pads, you can insert the puck into the sample chamber by using the instructions below.

1. Disengage the puck-insertion (puck-extraction) tool² by flipping up the black switch located on top of the tool or by fully depressing the switch, as shown in Figure 4-12.



Figure 4-12. Handle of puck-insertion (puck-extraction) tool, disengaged and engaged

- 2. Insert the puck, with the sample facing upward, into the hollow cylinder at the bottom of the puck-insertion tool. The sample will be inside the cylinder and the connectors and solder pads will be outside the cylinder (see Figure 4-13).
- 3. Rotate the puck to verify that it is properly seated inside the hollow cylinder of the puck-insertion tool. The puck should rotate smoothly.





¹ The P150 sample-wiring test station is also referred to as the puck-wiring test station.

² The name of this tool varies by context—it might be called the puck-insertion tool, the puck-extraction tool, or the sample-holder tool.

- 4. Engage the puck-insertion tool by flipping down the black switch located on top of the tool or by releasing the switch, if it is fully depressed, so that the switch lies flat across the handle (see Figure 4-13). The tool should now be gripping the outer rim of the puck.
- 5. Verify that the puck remains properly seated in the hollow cylinder of the puck-insertion tool. The puck must be level and it must not rotate—otherwise it could come loose in the sample chamber or bend the pins at the bottom of the sample chamber. If the puck lodges in the sample chamber, you might have to disassemble the probe to remove it.

CAUTION!

The puck-insertion tool (and puck) will be inserted into the sample chamber. Verify that the puck is level within the cylinder of the puck-insertion tool and firmly attached to the tool so that it cannot fall into the sample chamber. Also, hold the tool so that the bottom is level when you insert it into the sample chamber. If the puck lodges in the sample chamber, you might have to disassemble the probe to remove it.

6. Verify that the temperature of the sample chamber is at or above 298 K. The temperature *must* be at least 298 K when the chamber is opened to the atmosphere to prevent cryopumping air into the chamber. If the temperature of the chamber is below 298 K, set it to 298 K and wait until it reaches room temperature.

You can set the temperature by using the CTRL >> 3. Immediate Operations >> 1. Temp menu in the Model 6000, by using the shortcut in the MultiVu Status bar, or by using MultiVu.

CAUTION!

Always bring the sample chamber to room temperature before you open it to the atmosphere. This will prevent condensation and cryopumping of air constituents inside the chamber, which can cause probe malfunctions such as blocked valves and loss of temperature control.

 Verify that the field in the magnet is less than 1 tesla. If the field is greater than that, set the field to less than 1 tesla and wait for the magnet to reach the set point (select Instrument >> Field to open the MultiVu Field dialog box).

CAUTION!

Do not place the puck-insertion tool (or any other object) into the sample chamber when there are high fields in the magnet, as the force on the insertion tool could overwhelm you and cause you to damage the equipment.

8. Vent the sample chamber with clean, dry gas. Venting helps keep the sample chamber free of contaminants from the air.

To vent the chamber, you can use the Model 6000 menu (CTRL >> 1. Interactive Control >> 5. Vent Continuous), or you can use the PPMS MultiVu application software (Instrument >> Chamber >> Vent Cont.).

- 9. Open the hinge clamp and remove the KF blank flange from the sample-chamber access port (see Figure 2-5). If the blank flange is difficult to move because the internal pressure is low, *do not force it*. Allow the pressure within the chamber to match the external pressure before you open the sample chamber to atmosphere.
- 10. Remove the O-ring from the sample-chamber access port.
- 11. Gently lower the puck-insertion tool into the sample chamber with the puck-end first. Stop when the sample puck touches the puck connectors at the bottom of the chamber. *Do not* force the puck down farther after it touches the connectors.
- 12. Slowly rotate the puck-insertion tool until the key on the puck drops into the indexing notch. When the puck drops into the notch, you will feel it lock into position.
- 13. Gently push down on the puck-insertion tool in order to engage the puck interface and to make solid electrical contact between the interface and the puck.
- 14. Disengage the puck-insertion tool and then raise the tool several centimeters. Be alert for resistance when you raise the insertion tool. Resistance can indicate that the puck has caught in the tool as you began lifting it out, so you will need to remove the puck and again try to insert it.
- 15. Remove the puck-insertion tool from the sample chamber.
- 16. Place the O-ring over the sample-chamber access port and place the KF blank flange on it.
- 17. Place the flange clamp in position around the top of the sample-chamber access port and then latch the clamp.
- Purge and seal the sample chamber. To purge and seal the chamber, you can use the Model 6000 menu (CTRL >> 1. Interactive Control >> 2. Purge and Seal) or MultiVu (Instrument >> Chamber >> Purge/Seal).

The system is now prepared for you to conduct experiments. To determine your next steps, refer to the appropriate option manual. After you have performed a measurement and verified the operations of the instrument, you might find it useful to write a sequence that automates the measurement, as explained in the *Physical Property Measurement System Commands Manual* and the *Physical Property Measurement System: PPMS MultiVu Application User's Manual*.

4.5.2 Removing a Sample Puck

The procedures for removing the puck from the sample chamber are essentially the reverse of the installation procedure.

1. Verify that the sample chamber is at or above 298 K. The temperature must be at least 298 K to prevent cryopumping of air into the chamber. If the temperature is below 298 K, set it to 298 K and wait for the chamber to reach room temperature.

CAUTION!

Always bring the sample chamber to room temperature before you open it to the atmosphere. This will prevent condensation and cryopumping of air constituents inside the chamber, which can cause probe malfunctions such as blocked valves and loss of temperature control.

2. Verify that the field in the magnet is less than 1 tesla. If the field is greater than that, set it to less than 1 tesla and wait for the magnet to reach the set point.

CAUTION!

Do not place the puck-extraction/insertion tool³ (or any other object) into the sample chamber when high fields are in the magnet, as the force on the extraction tool could overwhelm you and cause you to damage the equipment.

3. Vent the sample chamber with clean, dry gas to help keep the sample chamber free of contaminants in the air.

To vent the chamber, you can use the Model 6000 menu (CTRL >> 1. Interactive Control >> 5. Vent Continuous) or you can use the PPMS software (Instrument >> Chamber >> Vent Cont.).

- 4. Open the hinge clamp and remove the KF blank flange from the sample-chamber access port (see Figure 2-5). If the blank flange is difficult to move due to low internal pressure, do not force it. Allow the pressure within the chamber to match the external pressure before you open the sample chamber to atmosphere.
- 5. Remove the O-ring from the sample-chamber access port.
- 6. Disengage the puck-insertion tool by flipping up the black switch located on top of the tool or by fully depressing the switch (see Figure 4-12).
- 7. Gently lower the puck-insertion tool, cylinder-end first, into the sample chamber until the tool touches the bottom of the chamber.
- 8. Engage the puck-insertion tool by flipping down the black switch located on top of the tool or by releasing the switch, if it is fully depressed, so that the switch lies flat across the tool's handle (see Figure 4-12).
- 9. Gently raise the insertion tool out of the sample chamber. You should feel some initial resistance as you pull the puck out of its seat.
- 10. Verify that the sample puck is in the insertion tool. If it is not, return to Step 6. If it is, disengage the lever and let the puck fall safely into your hand. *Do not drop the puck*.

Now you can insert another puck, install a PPMS option into the sample chamber, or close the sample chamber.

4.5.3 Closing an Empty Sample Chamber

Use the procedures below to close the sample chamber when it does not have a sample installed.

- 1. Place the O-ring and KF blank flange over the sample-chamber access port (see Figure 2-5).
- 2. Place the flange clamp in position around the top of the sample-chamber access port.
- 3. Latch the clamp.
- Purge and seal the sample chamber by using the Model 6000 menu (CTRL >> 1. Interactive Control >> 2. Purge and Seal) or MultiVu (Instrument >> Chamber >> Purge/Seal).

³ See Footnote 1.

4.6 System Customization

The PPMS is designed to be flexible and meet a variety of needs, so it accommodates customization. Before you begin any modifications, please read all applicable portions of the manual so that you understand how the components function and how your alterations could affect the system. Contact Quantum Design if you have questions about altering the system. Some common issues are addressed below.

4.6.1 Making Alternate Connections to the Sample Leads

If the Model 6000 does not provide the function you need, you can make electrical contact to the sample from other instruments by connecting an adapter to the gray Lemo connector cable. The pinouts are mapped for this purpose in Appendix A.

CAUTION!

Always use an adapter to access the leads in the gray Lemo connector cable (the cable that connects the probe head to the Model 6000). Use of an adapter ensures that you will be able to use the cable with other applications.

Figure 4-14 illustrates how to use an adapter to make connections at the "D" connector end of the gray Lemo cable. You can confine the wiring for this adapter to a box. We highly recommend using a breakout box, which provides an easily configured, reusable method of changing sample connections. It will keep the process flexible and isolate each wire in the gray Lemo cable, allowing contact to each wire individually. Such a box would have a female 25-pin "D" connector (DB-25) and input and output connections to the leads as necessary: for example, 12 banana plug connectors, 6 BNC connectors, other "D" connectors, or a bread board.



Figure 4-14. Custom adapter for making connections to the sample leads

4.6.2 Using Other Electronic Devices

The Model 6000 is designed to facilitate use with many other electronic instruments. It has an input for an external pressure gauge and an output to an external motor. Its four analog output BNC connectors monitor a variety of system signals. The "P8–Auxiliary" port contains two signal inputs to an analog-to-digital converter in order to digitize and record external signals. The "P1–User Bridge" port provides access to two current drivers, and the "P8–Auxiliary" port contains +15 V and -15 V low-power current sources. Several digital inputs and outputs are in various locations on the Model 6000. See Appendix A for detailed information about these capabilities.

You also can use the Model 6000 front panel to monitor additional equipment that uses the Model 6000 digital input and output lines. The state of each digital input and output is displayed in the **Digital:** status line at the bottom of the **Status–System Cont.** screen. The status codes are summarized in Table 4-3.

STATUS CODE	LINE AND STATE	TYPE OF LINE	LOCATION
HL	Hold Line Active (Low)	Busy Input	P–11 External
UR	User Line Active (Low)	Busy Input	P–11 External
A1	Auxiliary Drive #1 Active (-24 V)	Auxiliary Output	P–8 Auxiliary
A2	Auxiliary Drive #2 Active (-24 V)	Auxiliary Output	P–8 Auxiliary
LM	Limit Switch Active (+5 V)	Motor Input	P–10 Motor
NX	Index Switch Active (+5 V)	Motor Input	P–10 Motor
S1	Select Line #1 Active	Select Output	P–11 External
S2	Select Line #2 Active	Select Output	P–11 External
S3	Select Line #3 Active	Select Output	P–11 External
AC	Actuator Activated (-24 V)	Actuator	P–10 Motor

Table 4-3. Status codes for the Model 6000 digital inputs and outputs

Appendix A contains the information that is necessary for correctly interfacing other electronic devices with the PPMS. For example, some pressure gauges can be permanently damaged if they are connected improperly. Always verify that any electrical connections to the PPMS are solid and properly grounded.

4.6.3 Modifying the Gas and Vacuum System

If you plan to modify the gas and vacuum lines in the PPMS, first read Chapters 2 and 3 (Sections 3.3 and 3.4). Be especially careful that you understand how the PPMS functions, because your alterations can easily affect temperature control. When you make your changes, verify that any new plumbing connections are solid and have good seals.

4.7 Routine Maintenance Procedures

This section describes routine maintenance procedures that you should perform regularly. Less frequently performed procedures are discussed in Appendix B or Appendix C.

Regular maintenance procedures include adjusting the puck; transferring nitrogen and helium into a cold dewar, which you typically perform several times a week; basic servicing of the vacuum-pump assembly, which you should perform throughout the year; and O-ring inspections, which you should perform whenever you see an O-ring in the PPMS.

The less frequently performed procedures include transferring liquid nitrogen and helium into a warm dewar (Appendix B) and performing major pump-assembly services (Appendix C). Appendix C also contains a maintenance schedule and a form for tracking equipment service.

4.7.1 Puck Adjustment

You will need to adjust the sample puck whenever it fits loosely into the bottom of the chamber or after you have inserted it into the sample chamber approximately 10 times. Figure 4-15 displays the components of the puck-adjustment tool, and the steps below explain how to use it.

1. Place the puck on the finger spreader (see Figure 4-15).



Figure 4-15. Puck-adjustment tool

- 2. Remove the puck from the finger spreader.
- 3. Place the puck inside the finger contractor.
- 4. Press straight down on the puck and continue pressing until the puck is pressed completely into the finger contractor. When the entire chuck is in the contractor, the contractor evenly applies force to the outside of the fingers, pushing them inward. The contractor pushes the fingers—regardless of external wear or variations on the puck—so that they obtain their optimal location.
- 5. Remove the puck from the finger contractor.
- 6. Place the puck inside the test cutout. Verify that the puck fits easily but snugly in the test cutout.

4.7.2 Refilling a Cold Dewar



4.7.2.1 TRANSFERRING LIQUID NITROGEN

If there is any liquid helium in the PPMS dewar, you can use the procedures below to transfer liquid nitrogen into the nitrogen-jacketed dewar. However, if the dewar contains no liquid helium, you must use the "warm" dewar-fill procedures in Appendix B. The warm dewar-fill procedures are designed to prevent blockages in the impedance tube.

To facilitate the liquid-nitrogen transfer, we recommend that you review the process before you begin. If you are unfamiliar with these transfers, ask for help from someone who is familiar with the supply vessel.

- 1. At the PPMS dewar, prepare for the liquid-nitrogen transfer:
 - a. Remove the brass fittings from one of the two liquid-nitrogen fill ports by turning the larger fitting counter-clockwise until it comes off the dewar. This prevents the O-ring from freezing.
 - b. Open the other nitrogen fill port by turning the larger brass fitting counter-clockwise to loosen it and then removing the small insert plug when it is loose (see Figure 4-16).



NITROGEN FILL PORT

Figure 4-16. Preparing for a liquid nitrogen transfer

- 2. Screw the liquid-nitrogen transfer adapter onto the end of the nitrogen supply line.
- 3. At the PPMS dewar, insert the small end of the liquid-nitrogen transfer adapter into the open liquid-nitrogen fill port and turn the brass fitting clockwise to secure the adapter in place (Figure 4-16).

4. At the liquid-nitrogen supply dewar, slowly turn the liquid supply valve until it is about 50% open. Exhaust should begin coming out of the second nitrogen fill port on the PPMS dewar. Do not open the liquid supply valve more than 50%—a fully opened valve can produce violent spillovers of exhaust that are extremely hazardous and difficult to avoid.



WARNING!

Always open the liquid supply valve slowly, and only open it about 50%. Although the transfer is fast when the valve is completely open, such transfers are extremely hazardous.

5. At the PPMS dewar, visually monitor the exhaust from the second nitrogen fill port during the entire fill process. While you are monitoring the exhaust, put on your protective gear. This gear is necessary to prevent serious burns from the extremely cold fitting, supply line, and transfer adapter.

Do not leave the PPMS unattended during this step and always stand at least 0.5 m (1.5 ft.) from the exhaust plume.

- 6. At the liquid-nitrogen supply dewar, close the liquid supply valve when the exhaust turns to liquid, indicating that the jacket is full. The fill time will depend on the amount that was in the jacket when you started and how fast you perform the transfer. For example, jackets that are refilled twice a week take about 15 minutes to refill.
- 7. At the PPMS dewar, perform in sequence the steps below:
 - a. Remove the liquid-nitrogen transfer adapter: turn the brass fitting counter-clockwise and lift the transfer adapter out of the dewar.

In the event that the fitting and adapter are frozen together, you can use a warm air blower to accelerate the thawing process. Otherwise, you must wait until the parts thaw enough to be separated.

b. Close both nitrogen fill ports: re-install the brass fittings and turn the large brass fittings clockwise.

CAUTION!

Always re-install the fill-port fittings and/or O-rings onto the nitrogen fill ports after you have transferred liquid nitrogen into the dewar. These fittings prevent dangerous ice blockages in the fill ports.
4.7.2.2 TRANSFERRING LIQUID HELIUM



If there is any amount of liquid helium in the PPMS dewar, use the procedures described here to transfer helium into the dewar. If there is no liquid helium in the dewar, you must use one of the warm dewar fill procedures in Appendix B to prevent blockages in the impedance tube.

To facilitate the liquid-helium transfer, we recommend that you review the process before you begin. If you are unfamiliar with these transfers, ask for help from someone who is familiar with the supply vessel.

- 1. Bring the helium-supply dewar close to the PPMS dewar.
- 2. Verify that the proper adapters and extensions are installed on the helium transfer line (see Figure 4-17).

Important: The extensions perform an essential function: The input extension ensures that liquid can always enter the transfer line, even as the level of liquid in the storage dewar changes. A short extension is used on the output line to help reduce boil-off from the transfer. It is less cumbersome than the long one used for transfers into a warm dewar (see Appendix B).





- 3. At the liquid-helium supply dewar, perform in sequence the steps below, using Figure 4-18 for reference:
 - a. Vent the pressure from the supply dewar by slightly opening the gas-phase valve.
 - b. After the pressure has been reduced, close the gas-phase valve.
 - c. Open the liquid access port. This port is open only during the transfer.
 - d. Close the primary relief valve on the supply dewar. *This valve remains closed only during the transfer procedures*.



Figure 4-18. Arrangement for refilling a cold, nitrogen-jacketed dewar with liquid helium. Note the short output extension on the transfer line.

- 4. Using rubber or plastic tubing, connect a helium-gas cylinder to the gas-phase port on the liquid-helium supply dewar, as shown in Figure 4-18.
- 5. At the PPMS dewar, open one of the two helium fill ports on the probe head by pulling the entire fixture straight up (see Figure 4-19).
- 6. Slowly lower the input end of the transfer line into the supply dewar. Tighten the adapters that seal the transfer line to the liquid access port. Continue lowering the input end until an exhaust plume appears at the output end.
- 7. Insert the adapter at the output end of the transfer line into the PPMS dewar through the helium-fill port (refer back to Figure 4-18). Push the transfer line completely into the dewar.

Gas will begin flowing from the output adapter. *Point the output-adapter-exhaust-tube away from the evacuation valve and the rest of the dewar top.* The exhaust will be so cold it can freeze and damage parts, such as O-rings and sealed valves.



Figure 4-19. Opening a helium-fill port on the head of a PPMS probe (standard dewar shown)

CAUTION!

Point the output-adapter-exhaust-tube *away* from all hardware on the dewar and probe head. The extremely cold exhaust can damage parts, especially O-rings and sealed valves.

- 8. Verify that each adapter on the transfer line is properly seated so that it seals the transfer line.
- 9. At the liquid-helium supply dewar, open the gas-phase valve.
- 10. At the helium-gas cylinder, open the regulator to start transferring helium into the PPMS dewar
- Monitor the helium transfer with the Liquid Helium Fill Status dialog (in MultiVu select Utilities >> Helium Fill) or the Model 6000 (select CTRL >> 1. Interactive Control >> 0. Fill Dewar).
- 12. When the helium level reads 85–100%, close the regulator at the helium-gas cylinder.
- 13. At the liquid-helium supply dewar, reset the valves:
 - a. Close the gas-phase valve.
 - b. Open the primary relief valve.
- 14. Remove the transfer line and adapters from the liquid-helium supply dewar and the PPMS dewar.
- 15. At the PPMS dewar, close the helium-fill port on the probe head by reinserting the relief valve.
- 16. At the liquid-helium supply dewar, close the liquid access port (see Figure 4-18).
- 17. The liquid helium transfer is now complete. The helium-level meter will turn itself off when you exit the **Fill Dewar** screen or if the fill time exceeds 30 minutes.

4.7.3 Servicing the Vacuum-Pump Assembly

To help ensure that your equipment is in working condition when you want to perform measurements, it is essential that you regularly maintain the PPMS vacuum-pump assembly. A rotary-vane pump, which is located inside the electronics cabinet (Figure 4-20), uses oil to help pull the vacuum. Oil mist is naturally expelled from the pump exhaust and collected by an oil-mist

filter installed on the inside wall of the electronics cabinet. Air to the pump is filtered through the foreline trap.

Maintenance Schedule

The pump-oil level, oil-mist filter, and foreline trap require regular maintenance using the procedures in Sections 4.7.3.1 and 4.7.3.2.

- At least once a month, check the oil level in the pump and and oil-mist filter and add oil as appropriate.
- At least once a week, check the amount of oil that has collected around the oil-mist filter and empty as necessary. Check the oil levels more frequently if the pump is heavily used.



Figure 4-20. Electronics cabinet and rotary-vane pump

- Once a year, the pump and the oil-mist filter cartridge need a complete service, as explained • in Appendix C.
- Twice a year, use the instructions in Appendix C to clean the foreline trap, which acts as the • inlet filter for the pump. Appendix C also contains a maintenance record so that you can track when service was last performed.

CAUTION!

Check the level of oil in the oil-mist filter at least once a week. If the filter becomes too full, oil can back up into the gas lines and plug the system.

Pump Versions

Since 1997, all PPMS units have used CE-compliant Edwards or Varian pumps; before that time, the PPMS was equipped with an Alcatel pump (see Figure 4-21).



Alcatel rotary-vane pump

Edwards rotary-vane pump

Varian rotary-vane pump Leybold rotary-vane pump

Figure 4-21. Vacuum (rotary vane) pumps used with the PPMS

Table 4-4 lists the characteristics of each pump and Sections 4.7.3.1–4.7.3.2 explain how to check the oil level in the pump and oil-mist containers. Appendix C contains the instructions for changing the oil and oil-mist filter cartridge and for servicing the foreline trap. For detailed information about your pump, refer to the separate vacuum-pump manual that comes with the system.

Римр	MANUFACTURER			
CHARACTERISTIC	Alcatel	Edwards	Varian	Leybold
Color	Orange and black	Gray	Ivory and gray	Gray with Side-car
Oil Type	Alcatel 100 Direct Drive Mechanical Vacuum Pump Fluid	Edwards Supergrade "A" oil	Varian General Purpose Mechanical Pump Fluid	Leybold HE-200
Oil Level	Between upper and lower markings, best at 1/2 or more	Between "max" and "min"	Between arrows, best at 1/2 or more	Between upper and lower markings, best at 1/2 or more
Oil-fill cap	Top foremost cap	Top, front right	Top, front left	Top, Center
Drain plug	Lower plug	Bottom right	Bottom front center	Bottom right

4.7.3.1 CHECKING THE OIL IN THE PUMP

Weekly or sooner (depending on the amount of use), check the amount of oil that has collected in the bell jar that surrounds the oil-mist filter. If the filter becomes too full, oil can back up into the gas lines and plug the system. Monthly or sooner (depending on the amount of use), check the oil level in the pump, refill it to the full mark with the appropriate oil (see Table 4-4), and check the status of the filter cartridge.

- 1. Open the front panel of the electronics cabinet. Figure 4-20 illustrates the cabinet and pump.
- 2. Look at the oil-level window on the front of the pump. The oil level should be between the two outer markings (see Figure 4-21). Table 4-4 gives the specific level for each pump.
- 3. If the pump needs more oil, first check whether the oil needs to be changed. While the cabinet is open, compare the oil in the pump to clean oil.
 - a. If the pump oil is clean, use the instructions in Section 4.7.3.2 to add oil.
 - b. If the pump oil is dirty, use the instructions in Appendix C to drain the pump and replace the oil.
- 4. Check the amount of oil in the oil-mist filter and container.
 - a. If the container is half full, use the instructions in Section 4.7.3.2 to empty it, unless you also need to change the filter cartridge. In the latter case, use the instructions in Appendix C.
 - b. If the filter cartridge looks like it is full of oil, use the instructions in Appendix C to install a new one.

4.7.3.2 ADDING OIL AND DRAINING THE OIL-MIST CONTAINER



WARNING!

Put the system in shutdown mode and disconnect (but leave seated) the two metal pumping lines before you service the pump or related components. If there are leaks into the sample chamber and cooling annulus, ice can form and cause serious system malfunctions.

Prepare the PPMS for Pump Service

- 1. Place the PPMS in **Shutdown** mode (in MultiVu, select **Instrument >> Shutdown**). When you place the system in shutdown mode, it automatically seals the sample chamber, turns off the heaters, and restricts the flow-control valve.
- 2. Disconnect—but leave seated—the two metal pumping lines that come from the probe head. When the pumping lines are disconnected in this way the sample chamber and cooling annulus are sealed at the probe head.
- 3. Leaving the rest of the system components turned on, turn off the pump according to the instructions below. If the pump has been in operation, you might need to let it cool before you work on it.
 - a. Early PPMS units without a toggle switch on the pump—unplug the pump to turn it off. *Do not turn off the switch on the power strip*—this strip powers other system equipment in addition to the pump.
 - b. Recent PPMS units with a toggle switch on the back of the pump—turn off the toggle switch.
- 4. Open the console cabinet and hold your hand near the pump. If the pump is uncomfortably warm, let it sit until it has cooled before proceeding to the next section.

Fill the Pump

- 1. If the pump parts are difficult to access, slide the pump forward out of the electronics cabinet.
- 2. If the system has an Alcatel pump (Figure 4-21), remove the black faceplate that frames the oil-level window.
- 3. Remove the oil-fill cap on the top of the pump (see Table 4-4). Save the O-ring.
- 4. Fill the pump with oil to the top mark of the oil-level window (do not overfill).
- 5. Reinsert the O-ring and oil-fill cap.
- 6. If the system has an Alcatel pump, replace the faceplate that frames the oil-level window.

Drain the Oil-Mist Filter

- 1. Hold a container under the bottom of the bell jar and unscrew the plug.
- 2. When the oil has drained, pour the oil into a used-oil container.
- 3. Screw the plug back into the bell jar.

CAUTION!

Check the level of oil in the oil-mist filter at least once a week. If the filter becomes too full, oil can back up into the gas lines and plug the system.

Prepare the PPMS for Use

- 1. Slide the pump back into the electronics cabinet.
- 2. Turn the pump on and wait one minute so that the metal pumping lines can be evacuated. Verify that the pumping lines are seated in their connectors but not pressed in completely.
- 3. Reconnect the two metal pumping lines to the probe head.
- 4. Purge and seal the sample chamber.
- 5. Close the front door of the electronics cabinet.
- 6. The procedures are now complete. Please dispose of any used oil properly.

4.7.4 Using the Probe-Lifting Assembly

WARNING!

Always remove and insert the probe very slowly—raise it about one inch per minute. The probe could explode violently if it is removed from the dewar rapidly and there is a leak in the vacuum space.

You must use specific techniques to handle the probe, because it is fragile and easily damaged. Quantum Design provides a probe-lifting assembly (Figure 4-22) to help you move the probe in and out of the dewar. To prevent damage to the probe when it is out of the dewar, please use the probe-handling guidelines described in Section 4.2.1.

The steps below explain how to use the probe-lifting assembly. We recommend that you review the procedures before you begin, referring to Section A.4 and Figures 2-5, 4-19, and A-3 if necessary.

- 1. Open the helium-fill ports on the probe head by pulling the relief valves straight up (Figure 4-19).
- 2. Remove the hinge clamp from the sample-chamber access port (Figures 2-5 and A-3).
- 3. From the front of the probe (the side with the Quantum Design logo), slide the probe-lifting assembly onto the probe head so that the lifting assembly is underneath the sample-chamber access port but above the pressure-relief valve (Figure A-3).





- 4. Use the pin on the end of the chain to close the locking mechanism of the lifting assembly. *You must close the locking mechanism before you lift the probe.*
- 5. Attach the probe-lifting assembly to a hoist or a pulley, if necessary to lift or lower the probe.
- 6. When you have finished raising or lowering the probe, remove the assembly.

4.7.5 Inspecting O-Rings

You can increase the reliability and lifetime of the PPMS by maintaining the O-rings so they are always in good condition. The regularly accessible O-rings are on the top-plate assembly and on the helium and nitrogen fill-port fixtures. You also might see other O-rings when you perform maintenance and servicing.

All O-rings in the system should be clean and they should be lubricated with silicon vacuum grease. To ensure that the O-rings remain in prime condition, adopt the following habits.

- If you see an O-ring, visually inspect it.
- If an O-ring appears dirty, clean it with a clean, lint-free cloth (e.g., a Kimwipe).
- If an O-ring is dry, apply silicon vacuum grease to it.
- If an O-ring is cracked, replace it.
- If an O-ring is leaking, contact Quantum Design.

Connections, Ports, and Pinouts

A.1 Introduction

This appendix contains the following information:

- Section A.2 illustrates the connections between the system hardware components.
- Section A.3 illustrates and describes the ports on the rear panel of the Model 6000.
- Section A.4 illustrates and describes the ports on the rear of the PPMS probe head.
- Section A.5 contains pinout tables for all electrical ports.
- Section A.6 lists the recommended replacement fuse values.

A.2 System Connections

The proper connections between the probe head, Model 6000, Model 6700, and pump are shown in Figure A-1. Use the figure as a guide when you connect the components (eliminate reference to Model 6700 in the figure below).

0

0



Figure A-1. Connections for PPMS hardware: solid lines identify electrical connections and dashed lines identify gas/vacuum lines.

Electrical connections to the probe have color-coded Lemo connectors at the probe end. Three separate pumping lines are attached to the probe: a smaller metal hose that attaches to the sample chamber, a larger metal hose that connects to the cooling annulus, and a small white polypropylene hose that connects to the dewar.

Figure A-1 does not illustrate connection of a personal computer to the system, which is through the "P4–IEEE488" (GPIB) port on the rear of the Model 6000 (see Section A.3.4).

A.3 Model 6000 Rear Panel Ports

Figure A-2 illustrates the rear panel of the Model 6000. The upper half contains electrical connections. The lower half houses the system fuses and connections to the gas lines for temperature and pressure control. Some ports provide access to standard PPMS hardware and others allow custom configuration.



Figure A-2. Ports on rear panel of Model 6000

A.3.1 P1–User Bridge Port

If the system includes the Resistivity option, the "P1–User Bridge" port accesses the additional bridge board that is in the Model 6000. The "P1–User Bridge" port usually connects to the gray-ringed Lemo connector on the probe head and thus connects the user bridge board to the installed sample.

The optional resistance bridge board provides channels for four separate four-wire resistance measurements. In some models, the small round port next to the "P1–User Bridge" port provides parallel access to the fourth of these channels. The Model 6000 Status–Bridge screen displays the status of the user bridge channels. Use the CTRL >> 3. Immediate Operations >> 06: Bridge menu to control each bridge channel. To record the current and resistance of each channel, use the CTRL >> 3. Immediate Operations >> 11: Measure command.

The user bridge board also provides access to two additional current drivers of the Model 6000. These drivers, which provide up to 1 A or 20 W of current (whichever current limit is reached first), can be monitored with the **Status–System Cont.** screen. To monitor them, use the **CTRL** >> 3. Immediate Operations >> 08: DrvOut menu. To record the current and power through each driver channel, use the CTRL >> 3. Immediate Operations >> 11: Measure command.

A.3.2 P2–System Bridge Port

The "P2–System Bridge" port accesses (1) the two heater drivers for the heaters on the sample chamber and (2) the PPMS system bridge board that monitors the three system thermometers. The "P2" port connects to the black-ringed Lemo connector on the probe head.

The system bridge board is identical to the optional user bridge board, but is required for system thermometry. You cannot access three of the four system bridge channels. You can access the fourth channel, but it is reserved for some PPMS options that require their own thermometer. If you plan to use the PPMS for resistance measurements, Quantum Design recommends using one of the PPMS resistivity options, because the base PPMS is not configured for easy access to the fourth system bridge channel.

A.3.3 P3–Option Port

The "P3–Option" port accesses boards that are installed with certain PPMS options. For example, the AC Measurement System option and the AC Transport option use the AC board, which is installed behind the "P3–Option" port. For details about this port, refer to the appropriate option manual or contact your Quantum Design representative.

A.3.4 P4–IEEE488 Port

The "P4–IEEE488" port is the GPIB communications port for the Model 6000. The GPIB provides a standardized method of communication for all types of electronic instruments. Multiple GPIB-capable instruments can be connected in parallel. Use the **CONFIG** >> **3. IEEE-488 Setup** menu to configure the GPIB.

A.3.5 P5–RS232 Port

The "P5–RS232" port provides an RS-232 interface to the Model 6000. Personal computers or other devices with standard RS-232 ports can be connected to this port. Use the **CONFIG** >> 2. Serial Port Setup menu to configure the "P5–RS232" port.

A.3.6 P6–Dewar Port

The "P6–Dewar" port connects the Model 6000 to the helium-level sensor, the impedance heater, and the superconducting magnet persistence switch heater. The "P6–Dewar" port connects to the blue-ringed Lemo connector on the probe head.

A.3.7 P7–Magnet Port

The "P7-Magnet" port connects the Model 6000 to the "M1" port on the Model 6700.

A.3.8 P8–Auxiliary Port

The "P8–Auxiliary" port offers several auxiliary outputs and inputs. Table A-8 lists the specific pinouts for the "P8–Auxiliary" port.

A.3.8.1 THREE 0 V/-24 V AUXILIARY SIGNAL DRIVES

The "P8–Auxiliary" port has three 0 V/–24 V auxiliary signal drives that act as relays. -24 V is considered the asserted state. The auxiliary signal drives might be used, for example, to open and close valves in the system. The total current available to these three drives, the motor actuator, and motor phase leads is 2 A.

The auxiliary signal drives are controlled from the **CTRL** >> **3. Immediate Operations** >> **07: DigSet** menu, via GPIB or serial port input, or from within a sequence. The asserted state of auxiliary signal drives 1 and 2 is represented in the **Status–System Cont.** screen by the "A1" and "A2" digital status codes. These status symbols are for digital inputs and outputs. Do not confuse them with the analog inputs or analog outputs called "A1" and "A2" on the Model 6000 rear panel.

A.3.8.2 CONSTANT +15 V AND -15 V OUTPUTS

The "P8–Auxiliary" port has constant +15 V and -15 V outputs that can be used as low-current power sources. For example, the +15 V and -15 V outputs can be used to power operational amplifiers. The +15 V and -15 V lines draw directly on the Model 6000 power supply, so it is important that these leads are never shorted.

Up to 200 mA of total current is available from the +15 V and -15 V lines when the ACMS and AC Transport options are not installed or active. However, this current is shared with the AC board. When the AC board is driving relatively large alternating currents for the ACMS or AC Transport option, the current that is available at these outputs drops to 10 mA.

A.3.8.3 TWO SENSE LINES

The "P8–Auxiliary" port has two sense lines that are essentially digital on/off inputs that operate at TTL levels (5 V = inactive, 0 V [shorted] = active). To record the status of each sense line, use the **CTRL** >> **3. Immediate Operations** >> **11: Measure** >> **DigIn** command. The Model 6000 does not control based on the status of the sense lines. The sense inputs can be used during an experiment, for example, simply to indicate when a certain instrument in the system is operating.

A.3.8.4 TWO ANALOG SIGNAL INPUTS

The "P8–Auxiliary" port has analog signal inputs (-10 V to +10 V) that can be digitized and recorded by the Model 6000. The Model 6000 records the two analog inputs at a rate of approximately 2 Hz. To measure the signal from each of these inputs, use the **CTRL** >> **3**. **Immediate Operations** >> **11: Measure** >> **Sig1** (or **Sig2**). The signal can be manipulated like any other data signal—that is, placed into data files, graphed with PPMS software, linked to the analog outputs, and so on. The status of these signal inputs can be observed in the **Status–System Cont.** screen when it has been enabled with the **CONFIG** >> **5. Software** >> **1. User Preferences** menu.

A.3.9 P9–Pressure Port

The "P9–Pressure" port provides power to and a signal from an external pressure gauge, such as a Pirani or Baratron gauge. The pinouts can be configured to match specific models of gauges. For more information, refer to Table A-9 and the manual for the gauge of interest, or contact a Quantum Design representative for more information about how to configure the pinouts for a gauge.

To see the port configuration, use the **CONFIG** >> 6. Hardware >> 4. Pressure Sensor menu. When this menu displays a selection other than "internal," all system pressure information is rerouted to obtain information from the indicated gauge (or none at all), rather than from the internal solid-state silicon pressure sensor of the Model 6000. The pressure shown in the **Status–System** screen reflects this pressure information. To record the pressure reading, use the **CTRL** >> 3. Immediate Operations >> 11: Measure >> More command.

Up to 200 mA of total current is available from the +15 V and -15 V lines when the ACMS and AC Transport options are not installed or active. However, this current is shared with the AC board. When the AC board is driving relatively large alternating currents for the ACMS or AC Transport options, the current that is available at these outputs drops to 10 mA.

A.3.10 A1, A2, A3, and A4 Ports

The "A1," "A2," "A3," and "A4" ports are the analog outputs. These four BNC connectors can be linked to any of about 30 different PPMS parameters, such as temperature, magnetic field, user bridge board resistance, or motor position. This allows -10 V to +10 V feedback to other instruments or connection to chart recorders, oscilloscopes, and so on. The "A1" output is also connected in parallel to the signal channel 1 connector on the front panel of the Model 6000.

When you link parameters to the analog outputs, you must specify a value for both 0 V and +10 V so that the appropriate gain and offset are used. Each channel has an output impedance of 100 Ω . You can use the CTRL >> 3. Immediate Operations >> 12: Link menu to link the channels to measurable parameters. You can also use the CTRL >> 3. Immediate Operations >> 10: SigOut menu to configure the analog outputs to supply constant voltages. The Status–System Cont. screen displays the status of each analog output.

A.3.11 P10–Motor Port

If the system includes options that use a Quantum Design sample transport or rotator motor, the motor is connected to the "P10–Motor" port. If a sample transport or rotator motor is not installed, then the "P10–Motor" port can be used to drive a small, 12 V, external stepper motor.

A 0/-24 V actuator, identical to the auxiliary digital signal drives in the "P8-Auxiliary" port, is included in the "P10-Motor" port. When this actuator is active, the **Status-System Cont.** screen displays the "AC" digital status code. The total current available to the motor actuator, motor phase leads, and three auxiliary signal drives is 2 A.

The "P10–Motor" port also includes a TTL-level index switch and limit switch leads in addition to the four phase leads and actuator leads. Index and limit switches should normally be wired closed. When these circuits are broken, the **Status–System Cont.** screen displays the "LM" and "NX" digital status codes to indicate that the limit and index switches are active. This occurs whenever a motor is *not* connected to the Model 6000 or whenever an index or limit switch is tripped.

You can use the **Status–System** screen to monitor the motor position. Commands pertinent to the motor configuration, position control, and position measurement are listed below. For more detailed information about each command, refer to the *Physical Property Measurement System Commands Manual.*

CTRL >> 1. Interactive Control >> 6. Move to Index

CTRL >> 1. Interactive Control >> 7. Move

CTRL >> 3. Immediate Operations >> 03: Move

CTRL >> 3. Immediate Operations >> 07: DigSet

CTRL >> 3. Immediate Operations >> 11: Measure

CONFIG >> 6. Hardware >> 3. Position Configuration

A.3.12 P11–External Port

The "P11–External" port has three optically isolated outputs and two digital input busy lines that can help synchronize PPMS activity with other instruments. The select line outputs provide TTL levels. A nominal 10 k Ω resistor must be used on the collector with the emitter tied to ground. The select line outputs can be controlled by using the **ExtSet** command within a sequence or by using the **CTRL** >> **3. Immediate Operations** >> **09: ExtSet** menu. When the select lines are activated, the symbols "S1," "S2," and "S3" appear in the **Status–System Cont.** digital status line.

You must provide the busy (input) lines with +5 V. When the busy lines sense the 5 V difference between this voltage and the input line, the channel is in a released state. When the input line is also at 5 V (the input lead shorted to the +5 V lead), the channel enters a hold state. There are two busy lines: a *user* line and a *hold* line. The hold state of each line is indicated in the **Status–System Cont.** screen by "UR" and "HL" on the digital input status line, respectively. You can also use the **CTRL** >> **3. Immediate Operations** >> **11: Measure** command to read the status. Furthermore, when you use the **sync** command within a sequence, the hold state of the hold line pauses sequence execution. This function applies only to the hold line, not the user line.

A.3.13 Annulus Port

The "Annulus" port connects the annulus to the flow-control valve in the Model 6000. The longer, 3/8-inch stainless steel hose connects the "Annulus" port to the QC quick-connect fitting on the probe head.

A.3.14 Pump Port

The "Pump" port connects the Model 6000 gas lines to the vacuum pump that pumps on the sample chamber and the annulus. The shorter, 3/8-inch stainless steel hose attaches to the "Pump" port.

A.3.15 System Vacuum Port

The "System Vacuum" port provides direct access to the system pump, allowing the connection of other gas and vacuum lines and devices between the vacuum pump and flush valve. Under normal circumstances, a short hose connects the "Sample Pump-Out" port to the "System Vacuum" port, allowing the pump to pump directly on the sample space.

A.3.16 Sample Pump-Out Port

The "Sample Pump-Out" port accesses the sample chamber through the flush valve in the Model 6000. When the flush valve is open, the sample chamber is open to this port. Under normal circumstances, this port is directly connected by a short hose to the "System Vacuum" port, which is internally connected to the "Pump" port and consequently to the vacuum pump.

A.3.17 Sample-Space Port

The "Sample Space" port connects the sample space to the vent valve, the flush valve, and the gas lines in the Model 6000. The 1/4-inch stainless steel hose connects the "Sample Space" port to the QC quick-connect fitting on the probe head.

A.3.18 Sample Vent-Up Port

The "Sample Vent-Up" port accesses the sample space through the vent valve in the Model 6000. Under normal circumstances, a small hose directly connects the "Sample Vent-Up" port to the "Gas Source" port, providing helium gas from the dewar boil-off for venting and purging the sample chamber. If you want to vent and purge the chamber with another gas, simply disconnect this hose and connect an alternate gas source to the "Sample Vent-Up" port. In this case, the "Gas Source" port must be plugged.

A.3.19 Gas Source Port

The "Gas Source" port connects internally to the dewar port to provide a helium gas source for sample chamber venting and purging. A short hose usually connects the "Gas Source" port to the "Sample Vent-Up" port, directing helium from the dewar into the sample space through the vent valve in the Model 6000. If you use an alternate gas source, the "Gas Source" port must be plugged.

A.3.20 Dewar Port

The polypropylene tubing provided with the system connects the "Dewar" port to the small Ultra fitting on the probe head. This connection to the dewar serves two purposes: (1) It provides a gas source for venting and purging the sample chamber, and (2) it allows monitoring of the pressure differential across the impedance assembly. This second function is necessary to allow proper low-temperature operation and control, so it is important to keep this line connected, even when an alternate gas source is used for venting the sample chamber.

A.3.21 Syst 5A Fuse

The "Syst 5A" fuse is the fuse for the system heater and stepper motor power supply. Replace the fuse only with an equivalent 5 A, 250 V, time-delay fuse.

A.3.22 Aux 2A Fuse

The "Aux 2A" fuse protects the auxiliary relays and external motor ports. Replace the fuse only with an equivalent 2 A, 3 AG fuse.

A.3.23 Quench Heater Fuse

The "Quench Heater" fuse is required to operate the Magnet Reset option used by the Ultra Low Field option. Replace the fuse only with an equivalent 630 mA, 280 V, time-delay fuse.

A.3.24 Power Receptacle

The power cord connection for the Model 6000 is at the rear in the lower right corner. The power receptacle contains two fuses for the controller power supply. Replace these fuses only with equivalent 2 A, time-delay fuses (120 VAC power environments), or with equivalent 1 A, time-delay fuses in 220 VAC power environments, as noted on the back panel. You can use a screwdriver or other flat instrument to pry open the door covering these fuses. Verify that the power setting, which is visible through a window in the door, is correct for the power being used.

A.4 Probe-Head Ports

The ports on the rear of the PPMS probe head connect the probe hardware to the Model 6000 and Model 6700.



Figure A-3. Ports on rear of PPMS probe head

A.4.1 Helium-Fill Ports

You open the helium-fill ports by pulling the relief valves straight up, thus allowing access directly into the helium dewar for regular dewar fills. Each of the two helium-fill ports is fitted with a 1-psi pressure relief valve to allow pressure to be released before dangerous levels are reached. *Do not* tamper with these pressure-relief valves.

A.4.2 Sample-Chamber Access Port

The sample-chamber access port is used to move samples in and out of the sample chamber.

A blank flange with an O-ring seal normally covers the sample-chamber access port. A hinge clamp, which is provided with the system, holds the blank flange on the top of the port. Some PPMS options have hardware that attaches to the flange.

A.4.3 Sample-Chamber Pressure-Relief Valve

The sample-chamber pressure-relief valve prevents the buildup of dangerous pressures within the sample chamber. The valve extends from the back of the sample-chamber access port. **Do not** tamper with this pressure-relief valve.

A.4.4 Smaller Metal Hose Connector

The smaller, 1/4-inch metal hose connector attaches to the "Sample Space" gas port on the rear of the Model 6000. The 1/4-inch metal hose connector allows sample-space venting and evacuation.

A.4.5 Larger Metal Hose Connector

The larger, 3/8-inch metal hose connector attaches to the "Annulus" gas port on the rear of the Model 6000. The 3/8-inch metal hose connector allows the pump to pull helium through the impedance tube and annulus.

A.4.6 Hose Nipple

The hose nipple does not need to connect to anything. The hose nipple can let you access the helium in the dewar so that you can observe the helium boil-off rate. This gas line, however, passes through a 1/3-psi pressure relief valve first, so the dewar should not be pressurized with this port. You can attach a helium recovery unit to this port.

A.4.7 Ultra Fitting

The 1/4-inch Ultra fitting should be connected to the "Dewar" port on the rear of the Model 6000 with a 1/4-inch white polypropylene hose. This line allows the Model 6000 to monitor the pressure differential between the dewar and the annulus during low-temperature operations. Additionally, this line provides helium from the dewar boil-off for venting the sample space.

A.4.8 Magnet Connector (TCM or Red Lemo Connector)

The TCM connector (earlier model systems have a red-ringed Lemo connector) attaches to the two large, color-coded terminals on the rear of the Model 6700. The red- or blue-banded cable connects to the blue (+) terminal on the Model 6700, and the black (unbanded) cable connects to the black (-) terminal. These connectors are illustrated in Section A.5.7.

Important: Verify that the magnet polarity is correct by using Table A-7.

A.4.9 Black Lemo Connector

The black-ringed Lemo connector attaches to the "P2–System Bridge" port on the rear of the Model 6000. The black Lemo connector contains connections to the three system thermometers and the two sample-chamber heaters.

A.4.10 Blue Lemo Connector

The blue-ringed Lemo connector attaches to the "P6–Dewar" port on the rear of the Model 6000. The blue Lemo connector contains connections to the helium-level meter, impedance thermometer, impedance heater, and magnet persistence switch.

A.4.11 Gray Lemo Connector

The gray-ringed Lemo connector contains connections to the sample-puck connectors on the bottom of the sample chamber. The gray Lemo connector might connect to one of several ports. It might connect to the "P1–User Bridge" port for four-wire resistance measurements; to the "P3–Option" port for use with PPMS options, such as the ACMS option; or to other PPMS controllers, such as the Model 7100 AC Transport Controller or the Model 6500 PPMS Option Controller. You can access pins on the "P1–User Bridge" port for connection to instruments other than the Model 6000. A list of pinouts is in Section A.5.

A.5 Pinout Tables

The following tables detail the pinouts for the electrical ports in the PPMS, including ports on the Model 6000, probe head, and sample puck. None of these tables includes pinout information for the "P3–Option" port on the Model 6000. Refer to the appropriate option manual for the pinout information regarding this port.

Note: The diagrams that accompany the tables illustrate hardware ports, not connectors at the end of the cables.

A.5.1 Sample Connections

The sample connector is at the bottom of the sample chamber. The gray Lemo connector is on the probe head. Pins 1 and 2 on the gray Lemo connector are connected to wires that extend into the annulus. These wires are not used for any function, but they provide a site for system expansion. The "P1–User Bridge" "D" connector is on the Model 6000.



Figure A-4. Sample connections: a) Sample puck, b) Gray Lemo connector, c) P1–User bridge port

SAMPLE PUCK	SAMPLE CONNECTO R	GRAY LEMO CONNECTO R	P1-USER BRIDGE "D" CONNECTOR	USER BRIDGE BOARD FUNCTION
			1	Cur Driver 1+ (unused)
			14	Cur Driver 1– (unused)
			2	Cur Driver 2+ (unused)
			15	Cur Driver 2– (unused)
3	3	3	5	Channel 1 I+
4	4	4	18	Channel 1 I–
5	5	5	6	Channel 1 V+
6	6	6	19	Channel 1 V-
7	7	7	7	Channel 2 I+
8	8	8	20	Channel 2 I–
9	9	9	8	Channel 2 V+
10	10	10	21	Channel 2 V-
11	11	11	9	Channel 3 I+
12	12	12	22	Channel 3 I–
13	13	13	10	Channel 3 V+
14	14	14	23	Channel 3 V–
			11	Channel 4 I+
			24	Channel 4 I–
			12	Channel 4 V+
			25	Channel 4 V-
			13	Shield

Table A-1. Sample connections

A.5.2 System Bridge Connections

The black Lemo connector is on the probe head. The "P2–System Bridge" "D" connector is on the Model 6000.



Figure A-5. System bridge connections: a) Black Lemo connector, b) P2–System bridge port

BLACK LEMO CONNECTOR	P2–SYSTEM BRIDGE "D" CONNECTO R	SYSTEM BRIDGE BOARD FUNCTION
8	3	Cur Driver Ch3+ (Block Heater)
7	16	Cur Driver Ch3– (Block Heater)
2	4	Cur Driver Ch4+ (Neck Heater)
1	17	Cur Driver Ch4– (Neck Heater)
14	5	Channel 1 I+
13	18	Channel 1 I–
12	6	Channel 1 V+
11	19	Channel 1 V-
6	7	Channel 2 I+
5	20	Channel 2 I–
4	8	Channel 2 V+
3	21	Channel 2 V–
16	9	Channel 3 I+
15	22	Channel 3 I–
9	10	Channel 3 V+
10	23	Channel 3 V–
	11	Channel 4 I+
	24	Channel 4 I–
	12	Channel 4 V+
	25	Channel 4 V-
	13	Shield

Table A-2. System bridge connections

A.5.3 Communication Port Connections (GPIB)

The "P4–IEEE488" port is on the Model 6000.

P4-IEEE488 port (GPIB)



Figure A-6. GPIB communication port connections

P4–IEEE488 CONNECTOR	GPIB FUNCTION
1	DIO1
2	DIO2
3	DIO3
4	DIO4
5	EOI
6	DAV
7	NRFD
8	NDAC
9	IFC
10	SRQ
11	ATN
12	SHLD
13	DIO5
14	DIO6
15	DIO7
16	DIO8
17	REN
18	GND
19	GND
20	GND
21	GND
22	GND
23	GND
24	GND

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A.5.4 Communication Port Connections (RS-232)

The "P5–RS232" port is on the Model 6000.

P5-RS232 port



Figure A-7. RS-232 communication port connections

P5-RS232 CONNECTOR	RS-232 FUNCTION
1	DCD
2	RXD
3	TXD
4	DTR
5	GND
6	DSR
7	RTS
8	CTS
9	TRIG

Table A-4. Communication port connections (RS-232)

A.5.5 Dewar Connections

The blue Lemo connector is on the probe head. The "P6–Dewar" port is on the Model 6000.





b) P6-Dewar port



Figure A-8. Dewar connections: a) Blue Lemo connector, b) P6-Dewar port

Table	A-5	Dewar	connections
Iable	A-J.	Dewai	COLINECTIONS

BLUE LEMO CONNECTOR	P6–DEWAR "D" CONNECTOR	FUNCTION
11	1	Level Meter +I
12	14	Level Meter –I
1	2	Level Meter +V
2	15	Level Meter –V
7	3	Impedance Htr Drv
8	16	Impedance Htr Rtn
9	4	Impedance Therm +
10	17	Impedance Therm –
15	5	Persist Switch Drv
16	18	Persist Switch Rtn
3	6	Mag Quench Htr (AC)
4	19	Mag Quench Htr (AC)
5	7	Magnet V+
6	20	Magnet V-
17	8	Magnet Trim +
18	21	Magnet Trim –
13	9	Special Function +
14	22	Special Function –
	10	Dewar Spare A
	23	Dewar Spare B
	11	Dewar Spare C
	24	Dewar Spare D
	12	Dewar Spare E
	25	Dewar Spare F
	13	Shield

A.5.6 Magnet Connections (Model 6700 to Model 6000)

The "M1" connector is on the Model 6700. The "P7-Magnet" port is on the Model 6000.





M1 "D" CONNECTOR	P7-MAGNET "D" CONNECTOR	FUNCTION
1	1	Digital Gnd
2	2	Data Out
3	3	Sys Sync
4	4	Reset
5	5	Return (15 V)
6	6	–24 V
7	7	Magnet V+
8	8	Shield
9	9	Clock
10	10	Magnet Sel
11	11	Data In
12	12	+15 V
13	13	–15 V
14	14	Return (-24 V)
15	15	Magnet V-

Table A-6. Magnet connections (Model 6700 to Model 6000)

A.5.7 Magnet Connections (Probe to Controller)

The magnet connector is on the probe head. Recent systems use a TCM connector and earlier systems use a red Lemo. The magnet current port is on the Model 6700.



a-2) Red Lemo connector



b) Magnet controller



Figure A-10. Magnet connections: a-1) TCM (100 A Top plate feedthrough) connector or a-2) Red Lemo connector and b) Magnet controller

Table A-7. Magnet connections	s (probe to controller)
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MAGNET CONNECTION	CONNECTO R	MAGNET CURRENT	FUNCTION
Red Lemo	1	Mag + (red- or blue-banded cable)	Current +
	2	Mag – (black cable)	Current –
TCM (100 A Top plate feedthrough)	1	Mag + (blue-banded cable)	Current +
	2	Mag – (black cable)	Current –

A.5.8 Expansion Connections (Auxiliary)

The "P8–Auxiliary" port is on the Model 6000.

P8–Auxiliary port





Table A-8. Expansion connections (auxiliary)

P8–AUXILIARY "D"	FUNCTION
CONNECTOR	
1	Aux Drive 1
14	Aux Rtn 1
2	Aux Drive 2
15	Aux Rtn 2
3	Aux Drive 3
16	Aux Rtn 3
4	+15 V Out
17	–15 V Out
5	15 V Com
18	Sense 1
6	Sense 2
19	Sense Gnd
7	Sig In 1
20	Sig In 2
8	Sig In Gnd
21	Spare 1
9	Spare 2
22	Spare 3
10	Spare 4
23	Spare 5
11	Spare 6
24	Spare 7
12	Hi Vac Solenoid
25	Hi Vac Solenoid
13	Shield

A.5.9 Expansion Connections (Pressure Gauge)

The "P9–Pressure" port is on the Model 6000.

P9–Pressure port





P9-PRESSURE "D" CONNECTOR	GAUGE AND FUNCTION
1	Pirani Sig
6	Pirani Rtn
2	Baratron Sig
7	Baratron Rtn
3	+15 V
8	–15 V
4	Pwr Common
9	NC
5	NC

Table A-9. Expansion connections (pressure gauge)

A.5.10 Expansion Connections (Motor)

The "P10–Motor" port is on the Model 6000.

P-10 Motor port



Figure A-13. Motor expansion connections

Table A-10. Expansion connections (mot	or)
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P10-MOTOR "D" CONNECTOR	FUNCTION
1	Pos Ph1
6	Pos Ph2
2	Pos Ph3
7	Pos Ph4
3	Act Drv
8	Act Gnd
4	Limit 1 (Limit)
9	Limit 2 (Index)
5	Limit Gnd

A.5.11 Expansion Connections (External)

The "P11–External" port is on the Model 6000.



Figure A-14. External expansion connections

P11-EXTERNAL "D" CONNECTOR	FUNCTION
1	Select 1 (C)
6	Select 1 (E)
2	Select 2 (C)
7	Select 2 (E)
3	Select 3 (C)
8	Select 3 (E)
4	+5 V In
9	Busy 1 In (Hold)
5	Busy 2 In (User)

Table A-11. Expansion connections (external)

A.6 Replacement Fuse Values

Tables A-12 and A-13 list the manufacturers' recommended replacement fuse values. The Quantum Design stock number for each fuse is shown in the far-right column of each table.

HARDWARE	PORT	FUSE VALUE AND SIZE	QUANTUM DESIGN STOCK NUMBER
Model 6000	Pwr Entry Module	2 A, 5 \times 20 mm Delayed Acting	FD2A-20MM
	Sys	5 A, 1¼ in. Fast Acting	F35
	Aux	2 A, 1¼ in. Fast Acting	F2.0A
	Quench Heater	.63 A, 5 \times 20 mm Delayed Acting	FD.63A-20MM
50-A Magnetic Power Supply	Pwr Entry Module	5 A, 5 \times 20 mm Slow Blow	F5-20MMSB
100-A Magnetic Power Supply	Pwr Entry Module	10 A, 5 \times 20 mm Slow Blow	F10-20MMSB
ACMS	Pwr Entry Module	1 A, 250 V, (20 mm) Delayed Acting	FD1A-20MM
ACT	Pwr Entry Module	1 A, 250 V, (20 mm) Delayed Acting	FD1A-20MM
Option Controller	Pwr Entry Module	1 A, 5 \times 20 mm Fast Acting	F1-20MM
High Vacuum Cntr	Pwr Entry	3.15 A, 5 \times 20 mm Slow Blow	FD3.15A-20MM

Table A-13. Replacement fuse values for 200–240 VAC systems

HARDWARE	PORT	FUSE VALUE AND SIZE	QUANTUM DESIGN STOCK NUMBER
Model 6000	Pwr Entry Module	1 A, 5 \times 20 mm Delayed Acting	FD1A-20MM
	Sys	5 A, 1¼ in. Fast Acting	F35
	Aux	2 A, 1¼ in. Fast Acting	F2.0A
	Quench Heater	.63 A, 5 \times 20 mm Delayed Acting	FD.63A-20MM
50-A Magnetic Power Supply	Pwr Entry Module	2.5 A, 5 \times 20 mm Slow Blow	FD2.5-20MMSB
100-A Magnetic Power Supply	Pwr Entry Module	5 A, 5 \times 20 mm Slow Blow	FD5-20MMSB
ACMS	Pwr Entry Module	.5 A, 250 20 mm Delayed Acting	FD05A (.5A)
ACT	Pwr Entry Module	.63 A, 5 \times 20 mm Slow Blow	FD.63A-20MM
Option Controller	Pwr Entry Module	.5 A, 5 \times 20 mm Fast Acting	F.5-20MM
High Vacuum Cntr	Pwr Entry	3.15 A, 5 \times 20 mm Slow Blow	FD3.15A-20MM

Filling Warm Dewars

B.1 Introduction

This appendix contains the following information:

- Section B.2 presents an overview of helium and nitrogen transfers into a warm dewar.
- Section B.3 explains how to transfer helium and nitrogen into warm nitrogen-jacketed dewars.
- Section B.4 explains how to transfer helium into a warm standard dewar (no nitrogen jacket).

B.2 Helium and Nitrogen Transfers into Warm Dewars



The procedures in this appendix explain how to fill a "warm" dewar, which refers to situations in which the PPMS dewar has never been filled with helium or the dewar has been unused for an extended period of time and is dry. If there is any liquid helium in the dewar, use the routine transfer procedures described in Sections 4.7.2.

These warm-dewar-fill procedures include safeguards to prevent frozen contaminants from blocking the impedance tube—such a blockage would disable temperature control in the PPMS. To use these procedures, you must have installed the probe in the dewar and connected the system pumping lines and electrical lines, as shown in Figure A-1. The magnet and user bridge-

board connections are not necessary to perform a dewar fill, but they are needed to use the system fully.

If you are not experienced at transferring liquid helium, ask for help from someone who is familiar with the liquid-helium supply vessel. At the least, read over these instructions to familiarize yourself with the process and materials before you begin the helium transfer.

B.2.1 Nitrogen-Jacketed Dewars

There are two methods for refilling a warm nitrogen-jacketed dewar. The quickest method involves a simultaneous transfer of liquid nitrogen and liquid helium (Section B.3.1). The other method minimizes helium loss during the transfer by using a sequential transfer of liquid nitrogen and liquid helium (Section B.3.2). The sequential procedure requires a continuous flow of helium gas through the probe impedance tube during the liquid-nitrogen transfer and for a subsequent 48 hours cooling period.

B.2.2 Standard Dewars

To fill a warm standard dewar (no nitrogen jacket), use the instructions in Section B.4.

B.2.3 Materials

- Rubber or plastic tubing, 1–2 m
- A helium backfill adapter (sequential transfer only)
- A helium transfer line with input extension and long output extension, an output adapter, and an input adapter (see Figure B-1)





- A liquid-helium supply dewar for filling the dewar, 100 liters, except systems with magnets, 14 T and above, which need 200 liters.
- A helium-gas cylinder (standard size) to pressurize the liquid-helium supply dewar or to provide a helium backfill. For systems with an EverCool dewar, this cylinder is in addition to the helium-gas supply cylinder that is used to replenish the system.
- A liquid-nitrogen supply dewar (nitrogen jacketed dewars only), about 80 liters.
- A liquid-nitrogen transfer adapter (included with the nitrogen-jacketed dewars)
- A liquid-nitrogen supply line (included with the nitrogen-jacketed dewars)
- An optional warm air blower for removing hardware (nitrogen-jacketed dewars only)
- Rags to wipe up liquids, such as condensed water from the air

Important: If you do not have a Quantum Design helium-transfer kit, your hardware might differ from the hardware described in this appendix.

B.3 Warm Fill: Nitrogen-Jacketed Dewars

WARNING!

Always wear protective clothing, including thermal gloves, eye protection, and covered shoes, when you work with liquid helium or any other cryogen. Review Section 1.4.1., "Cryogens, " before you transfer liquid helium.

B.3.1 Simultaneous Nitrogen and Helium Transfer

You can save time by filling the nitrogen jacket and helium dewar belly simultaneously. Note that more helium will boil away during a simultaneous transfer than during a sequential transfer (Section B.3.2), but a sequential transfer includes an 48 hours waiting period that is not needed for a simultaneous transfer.

The procedures for a simultaneous transfer are not difficult but involve many steps. To facilitate a trouble-free transfer, we recommend that you review the entire set of instructions before you begin. As noted in Section B.2.3, "Materials," you will be working with transfer lines and valves for three dewars (the PPMS dewar, a liquid-nitrogen supply dewar, and a liquid-helium supply dewar) as well as a helium-gas supply cylinder. The transfer setup is shown below in Figure B-2 (the liquid-nitrogen dewar is not shown). The entire process will be easier if there are two people.



Figure B-2. Arrangement for the simultaneous transfer of nitrogen and helium into a warm nitrogen-jacketed dewar. The supply line for liquid nitrogen is indicated at the left of the figure (nitrogen cylinder is not shown).

Set Up

3.

hoses:

- 1. Verify that the system pump and the Model 6000 are turned on and operating properly. If either the pump or the Model 6000 is turned off, turn it on. If either the pump or the Model 6000 is on but appears to be malfunctioning, contact a Quantum Design representative.
- Using the Model 6000 or the MultiVu Temperature dialog shown in Figure B-3 (Instrument >> Temperature), set the temperature to 5 K, the rate to 10 K/min, and the approach mode to fast settle.

By setting the temperature to 5 K, you open the flow-control valve and ensure the maximum flow through the impedance tube. Maximum flow is necessary to flush out the impedance and keep contaminants from freezing inside it while it cools to cryogenic temperatures. (The temperature will not actually drop to 5 K because there is no liquid helium in the dewar.)

At the PPMS dewar, prepare to attach the transfer



Figure B-3. **Temperature** dialog and settings for helium transfer

- a. Remove the brass fittings from one of the two liquid-nitrogen fill ports by turning the large fitting counter-clockwise until it comes off the dewar. This prevents the O-ring from freezing.
- b. Open the other nitrogen fill port by turning the large brass fitting counter-clockwise to loosen it and then removing the small insert plug when it is loose (see Figure B-4).


NITROGEN FILL PORT



- 4. Screw the liquid-nitrogen transfer adapter onto the end of the liquid-nitrogen supply line.
- 5. At the PPMS dewar, insert the small end of the liquid-nitrogen transfer adapter into the open liquid-nitrogen fill port and turn the brass fitting clockwise to secure the adapter in place. *Do not begin transferring nitrogen yet*—leave the nitrogen supply line closed.
- 6. Bring the liquid-helium supply dewar close to the PPMS dewar.
- 7. Verify that the proper adapters and extensions are installed on the helium transfer line (see Figure B-1). Note that the long extensions perform an essential function: The extension on the input line ensures that liquid can always enter the transfer line, even as the liquid level in the dewar changes. The extension on the output line forces liquid helium all the way to the bottom of the PPMS dewar, so that escaping cold gas will cool all the system components before it leaves the dewar.
- 8. At the liquid-helium supply dewar, set the valves and liquid access port:
 - a. Vent the pressure by slightly opening the gas-phase valve.
 - b. Close the gas-phase valve after the pressure has been reduced.
 - c. Open the liquid access port. This port is open only during the transfer.
 - d. Close the primary relief valve. This valve remains closed only during the transfer.
- 9. At the PPMS dewar, open one of the two helium fill ports on the probe head by pulling the entire fill-port fixture straight up (see Figure 4-19).
- 10. Simultaneously insert (a) the output end of the transfer line into the PPMS dewar through the open helium fill port and (b) the input end of the transfer line into the liquid-helium supply dewar through the liquid access port (see Figures B-1 and B-2).
- 11. Carefully lower both ends of the transfer line completely into the dewars and seat the adapters in their respective ports. Gas will begin flowing from the output adapter. Point the output-adapter-exhaust-tube *away* from all hardware on top of the dewar. The exhaust will get extremely cold and could damage some of the parts, especially O-rings and sealed valves.

CAUTION!

Point the output-adapter-exhaust-tube *away* from all hardware on the dewar and probe head. The extremely cold exhaust can damage parts, especially O-rings and sealed valves.

12. Verify that each adapter on the transfer line is properly seated and is sealing the transfer line.

- 13. Raise the input side of the transfer line about 1 cm (1/2 in.) off the bottom of the supply dewar so that it does not collect ice or other debris that might have settled on the bottom.
- 14. Using rubber or plastic tubing, connect a helium-gas cylinder to the gas-phase port on the liquid-helium supply dewar.

Transfer

- 1. At the liquid-helium supply dewar, open the gas-phase valve.
- 2. At the helium-gas cylinder, open the regulator and adjust the pressure to approximately 7 kPa (1 psi). This pressurizes the supply dewar and maintains positive flow from the supply dewar to the PPMS dewar.
- 3. Keep the dewar pressurized this way for 2 minutes before you perform Step 4.
- Use the Chamber dialog in MultiVu (select Instrument >> Chamber >> Purge/Seal) or the Model 6000 (CTRL >> 1. Interactive Control >> 2. Purge & Seal) to remove air from the sample chamber.
- 5. At the liquid-nitrogen supply dewar, open the liquid supply valve. Exhaust should begin coming from the second nitrogen fill port on the PPMS dewar.
- 6. At the PPMS dewar, visually monitor the exhaust from the second nitrogen fill port during the entire fill process. *Do not leave the PPMS unattended during this step, and always stand at least 0.5 m (1.5 ft) from the exhaust plume.*
- Monitor the helium transfer with the Liquid Helium Fill Status dialog (in MultiVu select Utilities >> Helium Fill) or through the Model 6000 (select CTRL >> 1. Interactive Control >> 0. Fill Dewar).
- 8. At the liquid-nitrogen supply dewar, close the liquid supply valve when the exhaust turns to liquid (this indicates that the nitrogen jacket is full). It will take about 1 hour to fill the jacket of a warm dewar, because most of the liquid nitrogen will evaporate until the jacket walls have cooled.

Note: The dewar belly will take slightly longer to fill than the nitrogen jacket, because helium has a lower boiling point than nitrogen. The helium-level reading will be negative until the helium in the dewar has reached the base of the helium-level meter.

9. When the helium level reads 30–40%, the impedance tube will no longer be exposed. You can then set a system temperature so that the PPMS will be ready to use when the transfer procedures have been completed.

CAUTION!

Let the liquid-helium level reading reach 30–40% before you change the PPMS set-point temperature.

Shut Off and Disassembly

- 1. When the liquid-helium level reaches 97–100% in the PPMS dewar (after about 1 hour), close the regulator at the helium-gas cylinder to stop the transfer.
- 2. At the liquid-helium supply dewar, reset the valves:
 - a. Close the gas-phase valve.
 - b. Open the primary relief valve.
- 3. Remove the helium transfer line and adapters from the liquid-helium supply dewar and the PPMS dewar.

- 4. At the PPMS dewar, close the helium fill port on the probe head by reinserting the relief valve.
- 5. At the liquid-helium supply dewar, close the valve on the liquid access port.
- 6. At the PPMS dewar, perform in sequence the steps below:
 - a. Put on your protective gear. This gear is necessary to prevent serious burns from the extremely cold fitting, supply line, and transfer adapter.
 - b. Remove the liquid-nitrogen transfer adapter by turning the brass fitting counterclockwise and lifting the transfer adapter out of the dewar.

In the event that the fitting and adapter are frozen together, you can use a warm air blower to accelerate the thawing process. Otherwise, you must wait until the parts thaw enough to be separated.

c. Close both nitrogen fill ports by reinstalling the brass fittings and turning the large brass fittings clockwise.

CAUTION!

Always re-install the fill-port fittings and/or O-rings onto the nitrogen fill ports after you have transferred liquid nitrogen into the dewar. These fittings prevent dangerous ice blockages in the fill ports.

7. The liquid-helium transfer is now complete. The helium-level meter will turn itself off when you exit the **Fill Dewar** screen or if the fill time exceeds 30 minutes.

B.3.2 Sequential Nitrogen and Helium Transfers

You can conserve liquid helium by cooling the system before you perform the helium transfer. To do this, you fill the liquid-nitrogen jacket and then let the system sit for 48 hours before you transfer in the liquid helium.

Important: When you transfer nitrogen and helium separately, you must maintain a continuous flow of helium through the impedance tube so that ice does not form within the tube. The procedures below will ensure a continuous helium flow.

The procedures for a sequential transfer are not difficult but involve many steps. To facilitate a trouble-free transfer, we recommend that you review the entire set of instructions before you begin. As noted in Section B.2.3, "Materials," you will be working with transfer lines and valves for three dewars (the PPMS dewar, a liquid-nitrogen supply dewar, and a liquid-helium supply dewar) and a helium-gas cylinder. The transfer setup is shown below in Figure B-5 (the liquid-nitrogen dewar is not shown). The entire process will be easier if there are two people.



Figure B-5. Arrangement for sequential transfer of nitrogen and helium into warm nitrogen-jacketed dewar, with the supply line for liquid nitrogen indicated at left of figure (nitrogen cylinder is not shown)

Set Up

- Verify that the system pump and the Model 6000 are turned on and operating properly. If 1. either the pump or the Model 6000 is on but appears to be malfunctioning, contact a Quantum Design representative.
- Using rubber or plastic tubing, connect a helium backfill adapter to a helium-gas cylinder, as 2. shown in Figure B-6. The helium backfill adapter is a fixture that fits into one of the helium fill ports on the probe head. The helium-gas cylinder provides the helium backfill during the liquid-nitrogen transfer and during the dewar cool-down period.
- Open one of the two helium fill ports on the probe head by pulling the relief valve straight 3. up, as shown in Figure 4-19.
- 4. Insert the helium backfill adapter into the helium fill port. Verify that the adapter fits snugly.





HELIUM GAS CYLINDER

PPMS DEWAR Figure B-6. Inserting a helium backfill adapter



- 5. At the PPMS dewar, prepare for the liquid-nitrogen transfer:
 - Remove the brass fittings from one of the two liquid-nitrogen fill ports by turning the a. large fitting counter-clockwise until it comes off the dewar (see Figure B-7). This will prevent the O-ring from freezing.
 - b. Open the other nitrogen fill port by turning the large brass fitting counter-clockwise to loosen it and then removing the small insert plug when it is loose.

- 6. Screw the liquid-nitrogen transfer adapter onto the end of the nitrogen supply line.
- 7. At the PPMS dewar, insert the small end of the liquid-nitrogen transfer adapter into the open liquid-nitrogen fill port and turn the brass fitting clockwise to secure the adapter in place.
- Using the Model 6000 or the MultiVu Temperature dialog shown in Figure B-3 (Instrument >> Temperature), set the temperature to 5 K, the rate to 10 K/min, and the approach mode to fast settle.

By setting the temperature to 5 K you open the flow-control valve to ensure the maximum flow through the impedance tube. Maximum flow is necessary to flush out the impedance and keep contaminants from freezing inside it while it cools to cryogenic temperatures. The temperature will not actually drop to 5 K because there is no liquid helium in the dewar.

- 9. At the helium-gas cylinder, prepare it to act as a backfill:
 - a. Open the regulator to allow helium gas into the dewar and through the impedance tube.
 - b. Adjust the regulator on the helium-backfill cylinder so that gaseous helium is expelled from the 1/3-psi relief valve (the hose nipple on the back of the probe head) and from the 1-psi relief valve (on the closed helium fill port).
 - c. Keep the dewar pressurized this way for 2 minutes before you proceed.
- Use the Chamber dialog in MultiVu (select Instrument >> Chamber >> Purge/Seal) or the Model 6000 (CTRL >> 1. Interactive Control >> 2. Purge & Seal) to remove air from the sample chamber.
- 11. At the helium-backfill cylinder, adjust the regulator so that helium is expelled from only the hose nipple. Place a wetted finger in front of the hose nipple to verify that helium is exiting there.

Nitrogen Transfer

- 1. At the liquid-nitrogen supply dewar, open the valve on the nitrogen supply line. Exhaust should begin coming from the second nitrogen fill port on the PPMS dewar.
- 2. At the PPMS dewar, visually monitor the exhaust from the second nitrogen fill port during the entire fill process. *Do not leave the PPMS unattended during this step, and always stand at least 0.5 m (1.5 ft.) from the exhaust plume.*

Important: Periodically check the flow from the helium-backfill cylinder by placing a wetted finger in front of the hose nipple on the probe head. If helium is not being released from the 1/3-psi relief valve behind this fixture, increase the flow of helium by adjusting the regulator of the helium-gas cylinder.

- 3. At the liquid-nitrogen supply dewar, close the liquid supply valve on the nitrogen-supplyline when the exhaust turns to liquid (this indicates that the jacket is full). It will take about 1 hour to fill the jacket of a warm dewar, because most of the liquid nitrogen will evaporate until the jacket walls have cooled.
- 4. At the PPMS dewar, remove the liquid-nitrogen transfer adapter and re-install the nitrogen fill-port fittings:
 - a. Put on your protective gear so that you do not receive serious burns from the extremely cold fitting, supply line, and transfer adapter.
 - b. Remove the liquid-nitrogen transfer adapter by turning the brass fitting counterclockwise and lifting the transfer adapter out of the dewar.

In the event that the fitting and adapter are frozen together, you can use a warm air blower to accelerate the thawing process. Otherwise, you must wait until the parts thaw enough to be separated.

c. Close both nitrogen fill ports by reinstalling the brass fittings and turning the large brass fittings clockwise.

CAUTION!

Always re-install the fill-port fittings and/or O-rings onto the nitrogen fill ports after you have transferred liquid nitrogen into the dewar. These fittings prevent dangerous ice blockages in the fill ports.

- d. Periodically hold a wetted finger in front of the hose nipple on the probe head to verify that the helium-backfill cylinder is still providing helium to the dewar.
- 5. Leave the system standing for 48 hours with a full nitrogen jacket and an active heliumbackfill cylinder.

Important: To avoid excessive helium loss, you should allow 48 hours for the dewar and probe to cool before you begin to transfer liquid helium.

Helium Transfer

- 1. Verify that the system pump and the Model 6000 are turned on and operating properly. If either the pump or the Model 6000 is turned off, turn it on. If either the pump or the Model 6000 is on but appears to be malfunctioning, contact a Quantum Design representative.
- Using the Model 6000 or the MultiVu Temperature dialog shown in Figure B-3 (Instrument >> Temperature), set the temperature to 5 K, the rate to 10 K/min, and the approach mode to fast settle.

By setting the temperature to 5 K, you open the flow-control valve and ensure the maximum flow through the impedance tube. Maximum flow is necessary to flush out the impedance and keep contaminants from freezing inside it while it cools to cryogenic temperatures. (The temperature will not actually drop to 5 K because there is no liquid helium in the dewar.)

- 3. Bring the liquid-helium supply dewar close to the PPMS dewar.
- 4. Verify that the proper adapters and extensions are installed on the helium transfer line (see Figures B-1 and B-5). Note that the long extensions perform an essential function: The long extension on the input line ensures that liquid can always enter the transfer line, even as the liquid level in the storage dewar changes. The long extension on the output line forces liquid helium to the bottom of the PPMS dewar so that escaping cold gas will cool all the system components before it leaves the dewar.
- 5. At the liquid-helium supply dewar, set the valves and liquid access port:
 - a. Vent the pressure by slightly opening the gas-phase valve.
 - b. After the pressure has been vented, close the gas-phase valve.
 - c. Open the liquid access port. This port is open only during the transfer.
 - d. Close the primary relief valve. *This valve remains closed only during the transfer procedure*.
- 6. At the helium-backfill cylinder, close the regulator.
- 7. At the liquid-helium supply dewar, insert the input end of the transfer line into the liquid access port (see Figures B-1 and B-5)
- 8. At the PPMS dewar, remove the helium backfill adapter from the helium fill port on the probe head. Then, quickly insert the output end of the transfer line into the PPMS dewar through the open helium fill port.
- 9. Carefully lower both ends of the transfer line completely into the dewars and seat the adapters in their respective ports. When gas begins to flow from the output adapter, point the output-adapter-exhaust-tube *away* from all hardware on top of the dewar. The exhaust will be extremely cold and it could damage some of the parts, especially O-rings and sealed valves.

CAUTION!

Point the output-adapter-exhaust-tube *away* from all hardware on the dewar and probe head. The extremely cold exhaust can damage parts, especially O-rings and sealed valves.

- 10. Verify that each adapter on the transfer line is properly seated and is sealing the transfer line.
- 11. Raise the input side of the transfer line about 1 cm (1/2 in.) off the bottom of the supply dewar so that it does not collect ice or other debris that might have settled on the bottom.
- 12. At the liquid-helium supply dewar, prepare for the transfer:
 - a. Connect the helium-gas cylinder to the gas-phase port.
 - b. Open the gas-phase valve.
- 13. At the helium-gas cylinder, open the regulator and adjust the pressure to approximately 7 kPa (1 psi).
- 14. Monitor the helium transfer with the Liquid Helium Fill Status dialog (in MultiVu select Utilities >> Helium Fill) or through the Model 6000 (select CTRL >> 1. Interactive Control >> 0. Fill Dewar). The helium-level reading will be negative until the helium in the dewar has reached the base of the helium-level meter.

Note: The dewar belly will take slightly longer to fill than the jacket, because helium has a lower boiling point than nitrogen.

15. When the helium-level meter reads 30–40%, the impedance tube will no longer be exposed. You can then set a system temperature so that the PPMS will be ready to use when the transfer procedures have been completed.

CAUTION!

Let the liquid-helium level reading reach 30–40% before you change the PPMS temperature set point.

Shut Off and Disassembly

- 1. When the helium level reaches 97–100% (after about 1 hour), close the regulator of the helium-gas cylinder to stop the transfer.
- 2. At the liquid-helium supply dewar, reset the valves:
 - a. Close the gas-phase valve.
 - b. Open the primary relief valve.
- 3. Remove the transfer line and adapters from the liquid-helium supply dewar and the PPMS dewar.
- 4. At the PPMS dewar, close the helium fill port on the probe head by reinserting the relief valve.
- 5. At the liquid-helium supply dewar, close the liquid access port.

The liquid-helium transfer is now complete. The helium-level meter will turn itself off when you exit the **Fill Dewar** screen or if the fill time exceeds 30 minutes.

B.4 Warm Fill: Standard Dewars



The procedures for a filling a warm standard (non-nitrogen jacketed) dewar with liquid helium are not difficult but involve many steps. Quantum Design staff strongly recommend that you read the entire set of procedures before beginning the transfer. As noted in Section B.2.3, "Materials," you will be working with transfer lines and valves for two dewars (the PPMS dewar and a liquid-helium supply dewar) and a helium-gas supply cylinder. The transfer setup is shown below in Figure B-8. Your preparation will facilitate the process, which also will be smoother if there are two people.



Figure B-8. Liquid-helium transfer arrangement for transferring helium into a warm non-jacketed dewar

Set Up

- 1. Verify that the system pump and the Model 6000 are turned on and operating properly. If either the pump or the Model 6000 is on but appears to be malfunctioning, contact a Quantum Design representative.
- Using the MultiVu Temperature dialog shown in Figure B-3 (Instrument >> Temperature) or the Model 6000, set the temperature to 5 K, the rate to 10 K/min, and the approach mode to fast settle.

By setting the temperature to 5 K you open the flow-control valve and ensure the maximum flow through the impedance tube. Maximum flow is necessary to flush out the impedance and keep contaminants from freezing inside it while it cools to cryogenic temperatures. Note

that the temperature will not actually drop to 5 K because there is no liquid helium in the dewar.

- 3. Bring the liquid-helium supply dewar close to the PPMS dewar.
- 4. Verify that the proper adapters and extensions are installed on the helium transfer line (see Figure B-1). Note that the extensions perform an essential function: The long extension on the input line ensures that liquid can always enter the transfer line, even as the liquid level in the storage dewar changes. The long extension on the output line forces liquid helium all the way to the bottom of the PPMS dewar, so that escaping cold gas will cool all the system components before it leaves the dewar.
- 5. At the liquid-helium supply dewar, set the valves:
 - a. Vent the pressure by slightly opening the gas-phase valve.
 - b. Close the gas-phase valve after the pressure has been reduced.
 - c. Close the primary relief valve. This valve remains closed only during the transfer.
- 6. Using rubber or plastic tubing, connect a helium-gas cylinder to the gas-phase port on the liquid-helium supply dewar.
- 7. At the PPMS dewar, open one of the two helium fill ports on the probe head by pulling the entire fill-port fixture straight up (see Figure 4-19).
- 8. At the liquid-helium supply dewar, open the liquid access port. *This port remains open only during the transfer.*
- 9. Simultaneously insert (a) the output end of the transfer line into the PPMS dewar through the open helium fill port and (b) the input end of the transfer line into the liquid-helium supply dewar through the liquid access port (see Figure B-8).
- 10. Carefully lower both ends of the transfer line completely into the dewars and seat the adapters in their respective ports. Gas will begin flowing from the output adapter. Point the output-adapter-exhaust-tube *away* from all hardware on top of the dewar. The exhaust will get extremely cold and could damage some of the parts, especially O-rings and sealed valves.

CAUTION!

Point the output-adapter-exhaust-tube *away* from all hardware on the dewar and probe head. The extremely cold exhaust can damage parts, especially O-rings and sealed valves.

- 11. Verify that each adapter on the transfer line is properly seated and sealing the transfer line.
- 12. Raise the input side of the transfer line about 1 cm (1/2 in.) off the bottom of the supply dewar so that it does not collect ice or other debris that might have settled on the bottom.

Transfer

- 1. At the liquid-helium supply dewar, open the gas-phase valve.
- 2. At the helium-gas cylinder, open the regulator and adjust the pressure to approximately 7 kPa (1 psi).
- 3. Keep the dewar pressurized this way for 2 minutes before you proceed.
- Use the Chamber dialog (in MultiVu select Instrument >> Chamber >> Purge/Seal) or the Model 6000 commands (CTRL >> 1. Interactive Control >> 2. Purge & Seal) to remove air from the sample chamber.
- 5. Monitor the helium transfer with the Liquid Helium Fill Status dialog (in MultiVu select Utilities >> Helium Fill) or through the Model 6000 (select CTRL >> 1. Interactive

Control >> **0. Fill Dewar**). The helium-level reading will be negative until the helium in the dewar has reached the base of the helium-level meter.

6. When the helium level reads 30–40%, the impedance tube will no longer be exposed. You can then set a system temperature so that the PPMS will be ready to use when the transfer procedures have been completed.

CAUTION!

Let the liquid-helium level reading reach 30-40% before you change the PPMS set-point temperature.

Shut Off and Disassembly

- 1. When the helium level reaches 97–100%, close the regulator at the helium-gas cylinder to stop the transfer. It takes approximately 1 hour to fill the dewar.
- 2. At the liquid-helium supply dewar, reset the valves:
 - a. Close the gas-phase valve.
 - b. Open the primary relief valve.
- 3. Remove the transfer line and adapters from the PPMS dewar and the liquid-helium supply dewar.
- 4. At the PPMS dewar, close the helium fill port on the probe head by reinserting the relief valve.
- 5. At the liquid-helium supply dewar, close the liquid access port.

The liquid-helium transfer is now complete. The helium-level meter will turn itself off when you exit the **Fill Dewar** screen or if the fill time exceeds 30 minutes.

Vacuum-Pump Assembly Maintenance

C.1 Introduction

This appendix contains the following information:

- Section C.2 provides an overview of the vacuum-pump assembly and its maintenance requirements.
- Section C.3 describes the three types of vacuum pumps provided with the PPMS.
- Section C.4 has instructions for changing the oil and the oil-mist filter cartridge in the rotary vane pump.
- Section C.5 has instructions for changing the activated alumina in the foreline trap.
- Section C.6 is a maintenance record.

C.2 Vacuum-Pump Assembly

The vacuum-pump assembly is located inside the electronics cabinet (see Figure C-1). It includes a rotary-vane pump that uses oil to help pull the vacuum, a foreline trap with activated alumina to filter the intake air, and an oil-mist filter that cleans the exhaust.



Figure C-1. PPMS electronics cabinet with front panel opened

For optimal performance of your system, the pump, oil-mist filter, and foreline trap require regular maintenance, as shown in Table C-1. Instructions for more major types of service are provided here, while instructions for basic services (e.g., adding oil to the pump) are in Chapter 4.

COMPONENT	SERVICE	FREQUENCY
Pump	Check oil level	Check monthly (sooner with heavy use)
	Add oil	When reaches lower part of oil-level window
	Change oil	When dirty, when vacuum is unsatisfactory, or yearly
Oil-mist filter	Dump oil	Check monthly and dump when half full or sooner
	Change cartridge	When saturated with oil
Foreline trap	Check activated alumina	Twice a year
	Change activated alumina	When discolored and yellowish

C.3 Pump Versions

The PPMS is generally equipped with one of three pumps: an Alcatel pump, an Edwards pump, or a Varian pump. Since 1997, all systems have used CE-compliant Edwards or Varian pumps. Figure C-2 shows the three pumps and Table C-2 lists some basic characteristics of each. For detailed information about your pump, refer to the separate vacuum-pump manual that was supplied with the system.



Alcatel rotary-vane pump

Edwards rotary-vane pump

Varian rotary-vane pump Leybold rotary-vane pump

Figure C-2. Versions of the PPMS vacuum pump

COMPONENT	OMPONENT				
	Alcatel	Edwards	Varian	Leybold	
Color	Orange and black	Gray	Ivory and gray	Gray with Side-car	
Oil Type	Alcatel 100 Direct Drive Mechanical Vacuum Pump Fluid	Edwards Supergrade "A" oil	Varian General Purpose Mechanical Pump Fluid	Leybold HE-200	
Oil Level	Between upper and lower markings, best at 1/2 or more	Between "max" and "min"	Between arrows, best at 1/2 or more	Between upper and lower markings, best at 1/2 or more	
Oil-fill cap	Top foremost cap	Top, front right	Top, front left	Top, Center	
Drain plug	Lower plug	Bottom right	Bottom front center	Bottom right	

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C.4 Changing the Pump Oil and Oil-Mist Cartridge

The pump oil must be changed once a year, unless it is dirty (compare it to clean oil) or the pump is not producing a satisfactory vacuum. In the latter cases, you should immediately change the oil, even if it has been less than a year since the last change. Check the oil-mist cartridge at the same time as the pump-oil levels and change it when it is full of oil. Use the same oil type that was provided with the equipment (see Table C-2).



WARNING!

Put the system in **Shutdown** mode and disconnect the two metal pumping lines before you service the pump or related components. If there are leaks into the sample chamber and cooling annulus, ice can form and cause serious system malfunctions.

C.4.1.1 PREPARE PPMS FOR SERVICE

- 1. Place the PPMS in **Shutdown** mode (in MultiVu, select **Instrument** >> **Shutdown**). When you place the system in **Shutdown** mode, the software automatically seals the sample chamber, turns off the heaters, and restricts the flow-control valve.
- 2. Disconnect—but leave seated—the two metal pumping lines that come from the probe head. When the pumping lines are disconnected the sample chamber and cooling annulus are sealed at the probe head.
- 3. Leaving the rest of the system components turned on, turn off the pump according to the instructions below. If the pump has been in operation, you might need to let it cool before you begin to work on it.
 - a. Early PPMS units without a toggle switch on the pump—unplug the pump to turn it off. *Do not turn off the switch on the power strip*—this strip powers other system equipment in addition to the pump.
 - b. Recent PPMS units with a toggle switch on the back of the pump—turn off the toggle switch.

4. Open the console cabinet and hold your hand near the pump. If the pump is uncomfortably warm, let it cool before you proceed to the next section.

C.4.1.2 DRAIN PUMP OIL

- 1. If the system has an Alcatel pump, remove the black faceplate that frames the oil-level window (Figure C-2).
- 2. Remove the oil-fill cap on the top of the pump. Save the O-ring.
- 3. Slide the pump forward, out of the cabinet, so the oil will be able to drain into a container.
- 4. Place an empty container (capacity at least one liter) under the drain plug on the front of the pump.
- 5. Remove the drain plug and allow the oil to drain completely, lifting the rear of the pump if necessary to empty it.
- 6. Reinstall the drain plug.

C.4.1.3 DRAIN (REPLACE) OIL-MIST FILTER CARTRIDGE

- 1. Unscrew the bell jar of the oil-mist filter, which is mounted on the inside wall of the electronics cabinet.
- 2. Pour the oil into the used-oil container.
- 3. Examine the filter cartridge. If the filter cartridge is not saturated with oil, go to Step 4 of this section. If the filter cartridge is saturated with oil, you must replace it (contact Quantum Design if you need a replacement).
 - a. Unscrew the oil-mist filter cartridge.
 - b. Lubricate and install the new O-ring (supplied with the cartridge) on the filter.
 - c. Lubricate and install the new seal (supplied with the cartridge) for the bell jar.
 - d. Screw on the new filter cartridge.
- 4. Screw the bell jar back into place.

C.4.1.4 FILL AND RE-INSTALL PUMP

- 1. Fill the pump with oil to the top mark of the oil-level window (do not overfill).
- 2. Reinstall the oil-fill cap.
- 3. If the system has an Alcatel pump, replace the faceplate that frames the oil-level window (Figure C-2).
- 4. Slide the pump back into the electronics cabinet.
- 5. Turn the pump on and wait one minute so that the metal pumping lines can be evacuated. Verify that the pumping lines are seated in their connectors but not pressed in completely.
- 6. Reconnect the two metal pumping lines to the probe head.
- 7. Purge and seal the sample chamber.
- 8. Close the front door of the electronics cabinet.
- 9. The oil-change procedure is now complete. Please dispose of the used oil properly.

C.5 Servicing the Foreline Trap

The foreline trap acts as the inlet filter for the pump. The filtering component is activated alumina, which needs to be checked twice a year.



WARNING!

Put the system in **Shutdown** mode and disconnect the two metal pumping lines before you service the pump or related components. Any leaks into the sample chamber and cooling annulus can produce ice and serious system malfunctions.

C.5.1.1 PREPARE PPMS FOR SERVICE

- 1. Open the front panel of the electronics cabinet (Figure C-1).
- 2. Place the PPMS in shutdown mode (in MultiVu, select **Instrument >> Shutdown**). When you place the system in shutdown mode, the software automatically seals the sample chamber, turns off the heaters, and restricts the flow-control valve.
- 3. Disconnect—but leave seated—the two metal pumping lines from the probe head. This seals the sample chamber and cooling annulus at the probe head.
- 4. Leaving the rest of the system components turned on, turn off the pump according to the instructions below. If the pump has been in operation, you might need to let it cool before you work on it.
 - a. Early PPMS units without a toggle switch on the pump—unplug the pump to turn it off. *Do not turn off the switch on the power strip*—this strip powers other system equipment in addition to the pump.
 - b. Recent PPMS units with a toggle switch on the back of the pump—turn off the toggle switch.
- 5. Open the console cabinet and hold your hand near the pump. If the pump is uncomfortably warm, let it cool before you proceed to the next section.

C.5.1.2 REMOVE ALUMINA CANISTER AND EXAMINE THE PELLETS

- 1. Carefully unscrew the cap on the front of the foreline trap (see Figure C-2). Note that there is a spring located on the shaft inside the canister.
- 2. Remove the activated alumina canister.
- 3. Hold the canister by the bottom (*not the edges*) and remove the items listed below, in order, from the top of the canister. (The bottom will fall out and spill the activated alumina if you hold the canister by the edges.)
 - a. long spring
 - b. wing nut
 - c. washer
 - d. lid
 - e. washer
 - f. short spring
 - g. grille

4. Examine the activated alumina pellets. If they are discolored and yellowish, replace them with fresh pellets. If you need replacement material, contact Quantum Design.

C.5.1.3 REASSEMBLE THE CANISTER, TRAP, AND PUMP

- 1. To reassemble the activated alumina canister, install the items in the order listed below (a reversal of the removal procedures):
 - a. grille
 - b. short spring
 - c. washer
 - d. lid
 - e. washer
 - f. wing nut
 - g. long spring
- 2. Insert the activated alumina canister into the foreline trap with the spring facing the opened end of the trap.
- 3. Screw the cap back onto the front of the foreline trap.
- 4. Turn on the pump and wait one minute so that the metal pumping lines can be evacuated.
- 5. Reconnect the two metal pumping lines to the probe head.
- 6. Purge and seal the sample chamber.
- 7. Close the front door of the electronics cabinet.

C.6 PPMS Vacuum-Pump Assembly Service Record

Use this service record to help schedule and track servicing of the vacuum-pump assembly (see Sections 4.7.4 and C.2–C.5. We provide two blank sheets for your convenience.

Pump Assembly

COMPONENT	MANUFACTURER	SERIAL NUMBER
Pump		
Foreline trap		

SERVICE TYPE	SERVICE DATE
Change rotary-vane pump oil	
Empty oil in oil-mist filter	
Change oil-mist filter cartridge	
Check activated alumina in foreline trap	
Change activated alumina in foreline trap	
Other (explain)	

COMMENTS

SERVICE TYPE	SERVICE DATE
Change rotary-vane pump oil	
Empty oil in oil-mist filter	
Change oil-mist filter cartridge	
Check activated alumina in foreline trap	
Change activated alumina in foreline trap	
Other (explain)	

COMMENTS

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