



Physical Property Measurement System

Dilution Refrigerator User's Manual

Part Number 1091-100, A5

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5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber

Foreign Patents

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

C O N T E N T S

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

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

P.2 Scope of the Manual

This manual discusses the Physical Property Measurement System (PPMS) Dilution Refrigerator option. This manual explains how to install the Dilution Refrigerator, discusses the hardware and software that are unique to the Dilution Refrigerator, and explains basic operation and maintenance procedures for the Dilution Refrigerator system.

P.3 Contents of the Manual

- Chapter 1 introduces the Dilution Refrigerator system and discusses the theory of operation for the Dilution Refrigerator system.
- Chapter 2 discusses the hardware used with the Dilution Refrigerator system.
- Chapter 3 discusses the Dilution Refrigerator system software.
- Chapter 4 describes the basic system operation of the Dilution Refrigerator system.
- Chapter 5 contains a troubleshooting guide and explains how to perform common maintenance procedures.
- Appendix A explains how to install the Dilution Refrigerator system.
- Appendix B illustrates Dilution Refrigerator system connections and contains pinout tables.

P.4 Conventions in the Manual

File menu	Bold text distinguishes the names of menus, options, buttons, and panels appearing on the PC monitor.
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.
.dat	The Courier font distinguishes characters you enter from the PC keyboard. The Courier font also distinguishes code and the names of files and directories.
Note	Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances.

CAUTION!

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



WARNING!

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

Theory of Operation

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the functions of the Dilution Refrigerator.
- Section 1.3 discusses the theory of operation for the PPMS Dilution Refrigerator.
- Section 1.4 describes temperature control and its operating modes.

1.2 Overview

The Quantum Design Dilution Refrigerator (Model P850), developed in collaboration with Oxford Instruments, Superconductivity, extends the minimum temperature of the Physical Property Measurement System (PPMS) to 0.05 K. The Dilution Refrigerator System is integrated seamlessly into the PPMS platform, and its operation is designed to be completely transparent to the PPMS measurement application software. Operation of the Dilution Refrigerator is very similar to operation of the PPMS Helium-3 System (P-825).

The Dilution Refrigerator (DR) is entirely self-contained. The refrigerator insert, pumping lines, electrical cables, and gas handling system are incorporated into a small cart that can be rolled away from the PPMS when not in use. The system is shipped from the factory charged with helium gas mixture sealed in the refrigerator. No leak checking of the system is required for operation, and a single room temperature o-ring at the PPMS top flange is the only seal that is made each time the system is cooled down.

The Dilution Refrigerator is compatible with the PPMS DR Heat Capacity option and user experiments. Once the sample is mounted on the refrigerator insert and installed in the PPMS sample chamber, the temperature control is handled automatically down to 0.05 K without the need to learn new measurement control software.

Note: For first time installations of the Dilution Refrigerator System, please refer to Appendix A.

1.2.1 System Hardware Requirements

The following components are required to run the Quantum Design Dilution Refrigerator System:

- PPMS Continuous Low-Temperature Control (CLT) option
- PPMS High-Vacuum option
- Model 1000 (Modular Control System)
- CAN Computer Interface Kit

1.2.2 System Hardware

The Model P850 Dilution Refrigerator system consists of the insert assembly, a gas handling system, a temperature control module, and a cart for storage. Sample mounting hardware allows user experiments to be mounted on the Dilution Refrigerator. In addition, a calorimeter included with the DR Heat Capacity Option can be mounted.

Details of the Dilution Refrigerator hardware are discussed in Chapter 2.

1.2.3 System Software

The software provided with the Dilution Refrigerator system consists of the Dilution Refrigerator Option installed in PPMS MultiVu, a diagnostic utility, and maintenance utilities. The Dilution Refrigerator Option must be installed in MultiVu in order for the Dilution Refrigerator to operate.

Details of the software architecture and use are discussed in Chapter 3.

1.3 Theory of Operation

The dilution refrigerator utilizes a mixture of ^3He and ^4He isotopes to cool to 50 mK. Below about 0.87 K, ^3He and ^4He liquid mixtures separate into two phases. One phase, commonly called the dilute phase, consists of mostly ^4He with about 6 % ^3He . The other phase, the concentrated phase, is almost pure ^3He . The interface between the two is referred to as the phase boundary. In a dilution refrigerator (see Figure 1-1), the mixing chamber is full of liquid, with the denser dilute phase on the bottom and the less dense concentrated phase on top. Typically, the phase boundary is near the vertical center of the mixing chamber. The dilute phase extends from the bottom of the mixing chamber through the counter-flow heat exchanger and partially fills the still. Pumping continuously on the still using a turbo pump backed by a diaphragm pump cools the still to about 0.6 K and reduces the ^3He concentration in the still. Osmotic pressure drives ^3He up the dilute side of the counter-flow heat exchanger from the mixing chamber into the still. In order to replace this ^3He in the dilute phase, ^3He atoms cross the phase boundary from the concentrated phase into the dilute phase. In this process, the ^3He atoms absorb heat, providing cooling power for the refrigerator.

Pumped ^3He gas exiting the diaphragm pump is circulated back into the condenser of the dilution refrigerator where it condenses into liquid. The PPMS sample chamber wall provides the cooling power for the condenser. The liquefied ^3He flows from the condenser, down through a flow

impedance into the still heat exchanger, and then down the concentrated side of the counter-flow heat exchanger, where the liquid is cooled before entering the mixing chamber. The liquid ^3He then crosses the phase boundary again, completing the circulation path. This circulation allows the dilution refrigerator to maintain a temperature of 50 mK continuously with no need to refill or use one-shot mode.¹

In order to isolate the cold dilution unit from the relatively warmed PPMS sample chamber, the sample chamber is maintained at high vacuum by the PPMS. In addition, a small amount of charcoal on the dilution refrigerator helps to ensure good high vacuum at the bottom of the sample chamber.

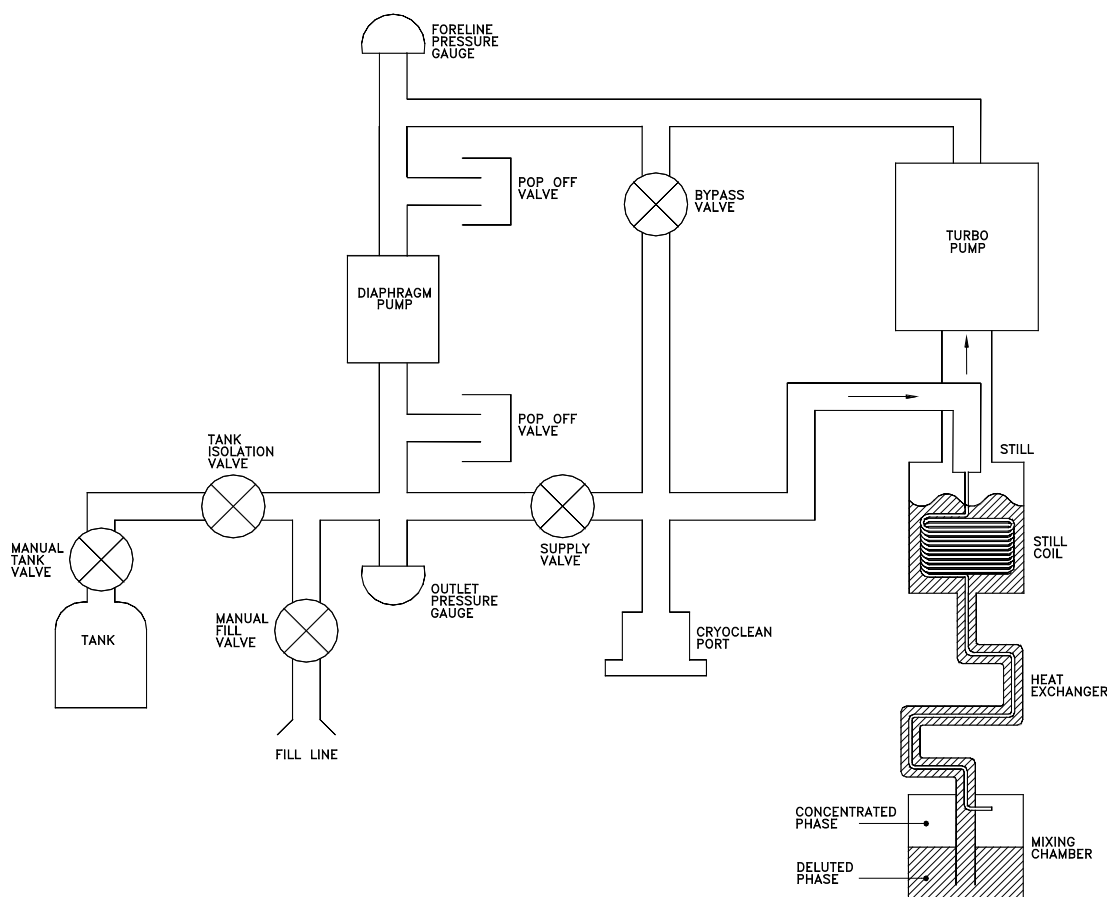


Figure 1-1: Functional diagram of the dilution refrigerator

Dilution cooling, described above, is used to cool the refrigerator at the lowest sample temperature, below 1 K. Between 1 K and 4 K, standard ^4He evaporative cooling is used. The different control modes are described in more detail in the next section.

¹ A more complete discussion of the operating principle of the dilution refrigerator can be found in F. Pobell. Matter and Methods at Low Temperatures. Berlin: Springer-Verlag, 1996.

1.4 Temperature Control

The Dilution Refrigerator is designed specifically to cool the sample below 0.5 K. However, because it can be inconvenient to remount samples for measurement up to higher temperatures, the system has been designed to allow temperature control from 50 mK to 4 K. Two distinct cooling modes are needed in order to achieve this range. In addition, the temperature set points between 295 and 300 K are used to warm the system to room temperature for removal of the probe from the PPMS. The Dilution Refrigerator Option software automatically controls the transitions between the modes as well as activates the PPMS High Vacuum system when needed. The operating modes are discussed in detail below.

1.4.1 Dilution Cooling (50 mK – 1.1 K)

In dilution cooling mode the system runs with the full ^3He - ^4He mixture in circulation with the helium liquid level in the still. The Bypass valve is closed while the Tank Isolation and Supply valves are open (see Figure 1-1). ^3He diffusing across the phase boundary in the mixing chamber produces the cooling power. Continuous circulation of ^3He is driven by the turbo pump, which runs at different speeds depending on the sample temperature set point. Returning mixture is reliquefied in the condenser, which is maintained at the PPMS base temperature, around 1.8 K. Control of the sample temperature is achieved by heating both the sample stage and the still. The still temperature set point is determined by the sample set point.

The system enters dilution cooling for set points below 1 K. Once in this mode, the system stays in dilution cooling mode for set points up to 1.1 K, creating some overlap and hysteresis with Evaporative Cooling mode.

Heat applied to a heater on the sample stage controls the sample temperature, measured by a thermometer integrated into the sample stage. A thermal standoff between the mixing chamber and sample stage allows the sample to be heated up to 1.1 K in this mode even though the mixing chamber must stay below about 0.6 K in order to maintain dilution cooling.

Before dilution cooling mode can be achieved, the mixture must first be condensed. During this time, the DR Console will display “Condensing for Dilution Cooling.” After condensing, the system may need to wait for dilution cooling to be established, during which time the DR Console displays “Waiting For Dilution Cooling.” Once dilution cooling has been established, the console displays “Dilution Cooling” and the system controls at the desired temperature set point.

1.4.2 Evaporative Cooling (1 K – 4 K)

In evaporative cooling the system runs with mostly ^4He in circulation with the helium liquid level down in the mixing chamber. The remainder of the mixture is isolated in the tank on the gas-handling system. The Bypass, and Tank Isolation valves are all closed while the Supply valve is open. Evaporative cooling takes place in the mixing chamber by pumping with the turbo pump through the heat exchanger. Returning mixture is reliquefied in the condenser, which is

maintained at 2.5 K by the PPMS sample chamber. Control of the sample temperature is achieved by heating both the sample stage and still.

The system enters evaporative cooling for set points between 4 K and 1 K. If the system is in dilution cooling mode, the system enters evaporative mode for set points between 4 K and 1.1 K.

In order to achieve evaporative cooling, the liquid ^3He must be distilled and stored in the tank so that nearly pure ^4He is left to circulate. During this distillation process, there will be a delay of about 1.5 hours before the desired sample set point is achieved. The DR Console will display “Distilling Mixture” during this time and the sample temperature will be held at 2 K. In addition, if the mixture has not already been condensed, the console will display “Condensing for Evap. Cooling” while the mixture is condensed.

Once the condensation and distillation process is complete, the console displays “Evaporative Cooling” and the system controls at the desired temperature set point.

1.4.3 High Temperature (4 K – 300 K)

Temperature set points between 295 and 300 K are used to warm the Dilution Refrigerator up from low temperature for removal of the insert from the PPMS. Set points between 4 K and 295 K are not allowed because the Dilution Refrigerator cannot control temperature in that range.

Below 290 K, the system is susceptible to developing plugs from gas contaminants in the condenser. In order to alleviate this problem the system is run with the Supply valve closed and the Bypass and Tank Isolation valves open in order to pump most of the gas from the insert. The control software does this automatically whenever the set point is below 295 K or the system is warming up and the PPMS has not yet reached 295 K.

The control software automatically pauses at 20 K during a cool down in order to activate high vacuum before cooling further. Also, the software automatically purges the sample space at 200 K during a warm up in order to aid warming of the dilution refrigerator insert, which would otherwise be isolated from the PPMS sample chamber by high vacuum.

1.4.4 DR Not Connected

When the gas handling system is not powered up and connected via CAN to the PC, the system is in “DR not connected” mode. In this mode, the MultiVu software behaves as if the Dilution Refrigerator option were not present and the PPMS sample chamber temperature is controlled in the normal way.

1.4.5 Probe-Out

The control software enters the “probe-out” mode when the gas handling system is powered up and connected to the PC but the probe is not inserted into PPMS. All valves are open and both pumps are off. In this mode both the PPMS and MultiVu software behave as if the Dilution Refrigerator option were not present. When the probe is inserted into the PPMS sample chamber, a micro-switch on the probe head is used to detect that the probe is in and the Dilution Refrigerator Option software takes over temperature control of the system.

1.4.6 DR Standby

The control software enters the DR Standby Mode when the control software detects that the probe has been inserted into the PPMS but no temperature set point has been received. All valves are open and both pumps are off. The system remains in this state until a temperature set point is received.

C H A P T E R 2

Hardware

2.1 Introduction

This chapter contains the following information:

- Section 2.2 discusses the overview of the Dilution Refrigerator system.
- Section 2.3 discusses the Dilution Refrigerator probe.
- Section 2.1 discusses the gas-handling assembly.
- Section 2.5 discusses the Model CM-D (Temperature Control Module).
- Section 2.6 discusses the Model 1000 (Modular Control System)
- Section 2.7 discusses CAN computer interface kit.
- Section 2.8 discusses the Electrical connections and cables.
- Section 2.9 discusses the Liquid nitrogen cold-trap assembly.
- Section 2.10 discusses the Option Specific hardware.
- Section 2.11 discusses other accessories included with the Dilution Refrigerator system.

2.2 Overview

The Dilution Refrigerator system consists of the probe and a gas-handling system permanently connected via a tether containing both vacuum and electrical connections. Additional electrical connections, used for temperature control and sample wiring are made at the head of the probe. Thermometry and heater electronics are housed in a separate temperature control module that plugs into the Model 1000 Modular Control System. Each piece of hardware is described in detail below.

2.3 Dilution Refrigerator Probe

2.3.1 Turbo Pump

A turbo pump is mounted at the head of the probe assembly to provide pumping for both dilution cooling and evaporative cooling modes. A diaphragm pump located on the gas-handling system backs the turbo pump. The speed of the turbo pump is varied to respond to changes in helium gas flow under different heat loads and operating modes. See Figure 2-1 for an overview of the dilution refrigerator probe.

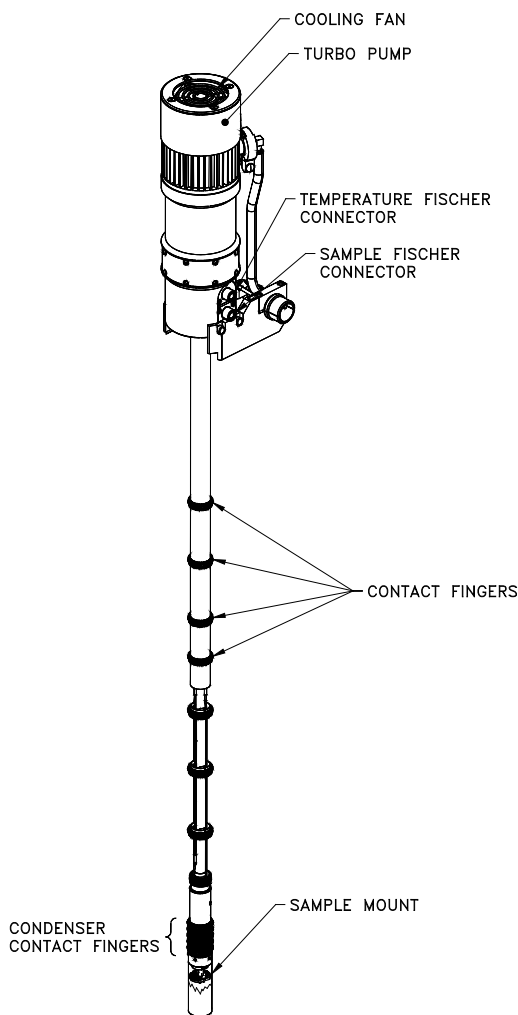


Figure 2-1. Dilution Refrigerator Probe

Mechanical vibrations can cause unwanted heating at milliKelvin temperatures. To prevent such vibrations the turbo pump on the Dilution Refrigerator insert is mounted atop a vibration-attenuating mechanism. The mechanism consists of a flexible stainless steel bellows connecting the turbo pump to the insert shaft. A black soft-rubber support surrounds the bellows and provides damping and structural support.

2.3.2 Thermal Contact Fingers

To provide the thermal isolation between the sample stage and the PPMS sample chamber necessary to reach milliKelvin temperatures, the PPMS sample chamber is maintained at a low pressure using the PPMS High-Vacuum option. While in high vacuum the majority of the probe is cooled through direct contact with the PPMS sample chamber. This is accomplished with seven rows of beryllium-copper contact fingers on the probe shaft and four rows of contact fingers on the condenser tube, which is threaded on to the condenser and surrounds the dilution unit. The four rows of fingers on the condenser tube contact the isothermal region of the PPMS sample chamber and provide cooling for the condenser. In this way, the condenser temperature can be controlled with the PPMS.

2.3.3 Dilution Unit and Sample Stage

The dilution unit and sample stage are located inside the condenser tube and radiation shield, and are comprised of the still, heat exchanger, mixing chamber and sample stage assembly (Figure 2-2). The circulating helium gas, mostly ^3He , enters the dilution unit through the condenser where it is cooled and condensed.

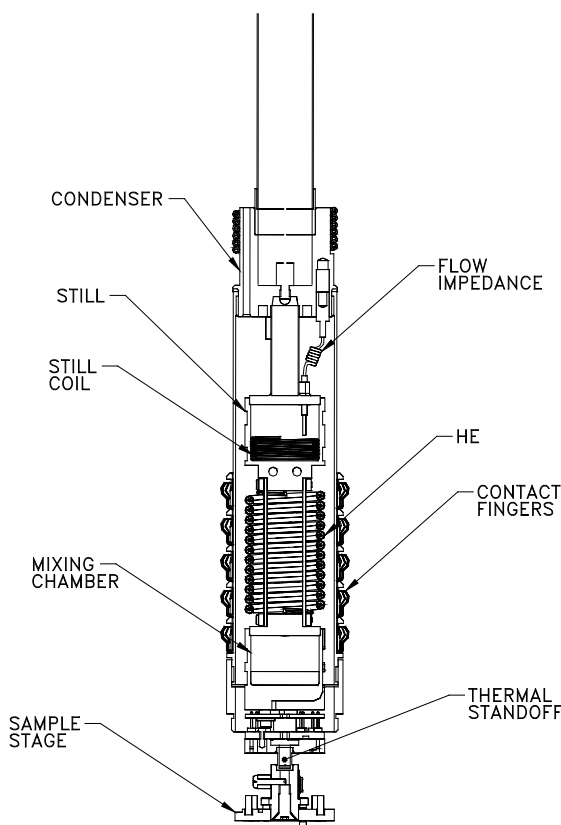


Figure 2-2. Dilution unit and condenser tube

The mixture then passes through the flow impedance into the still coil where it is further cooled. It next passes into the inner tube of the counter-flow heat exchanger, cooling the mixture further. The mixture enters the mixing chamber at the top, into the ^3He rich side of the mixture. Dilution

cooling occurs when ^3He from the rich phase diffuses across the phase boundary down into the dilute phase, consisting mostly of ^4He . A pumping tube, connected to the outer tube of the heat exchanger and the inside of the still, extends to the bottom of the mixing chamber. Osmotic pressure pushes the ^3He atoms in the dilute phase up the heat exchanger into the still. The low pressure in the still, produced by the turbo pump, causes the ^3He to evaporate, cooling the still. The mixture then runs through the gas handling system and back down to the condenser, completing the cycle.

The dilution unit, especially the small tubes of the counter-flow heat exchanger, is fairly delicate. In normal operation, the condenser tube and radiation shield protect the dilution unit from damage. When mounting and dismounting samples, one must take care not to damage the dilution unit. Use of the sample mounting pins, described in section 2.11, prevents damage to the dilution unit when mounting and dismounting samples.

CAUTION!

Due to the delicate nature of the dilution unit, you should not remove the condenser tube shield unless told to do so by Quantum Design Service personnel. Doing so could cause damage to the Dilution Refrigerator probe.

2.3.4 Probe Wiring

All of the wiring needed to run the Dilution Refrigerator and perform sample measurements is integrated into the refrigerator insert. The standard PPMS sample chamber wiring is not used and is not accessible on the Dilution Refrigerator. The probe wiring is separated into two looms, each connected to a separate Fischer connector at the probe head. The 12-pin Fischer connector, labeled TEMPERATURE, is used for system wiring and is connected to the sample thermometer, sample heater, still thermometer, and still heater. The 10-pin Fischer connector, labeled SAMPLE, is used for sample wiring, providing 8 leads for sample measurements. Pinout tables for the connectors and the sample stage can be found in Appendix B.

Heat leaks from wiring can significantly impact refrigerator performance. The wiring on the refrigerator insert is chosen to have low thermal conductance to minimize this heat leak. Also, the wiring is thermally anchored at the condenser, the still, and the mixing chamber before going to the sample stage in order to minimize the heat leak to each colder stage of the refrigerator.

WARNING!



Changing or adding to the wiring on the refrigerator insert can significantly impact the performance of the Dilution Refrigerator. Quantum Design recommends that you do not attempt to make any changes to the refrigerator insert wiring.

2.4 Gas-Handling System

The gas handling system contains all hardware necessary to circulate gas in the Dilution Refrigerator in order to achieve cooling. The gas handling system is connected to the refrigerator insert by a tether which contains two small gas lines and various electrical cables. The components of the gas-handling system are the diaphragm pump, mixture tank, gas manifold, turbo pump controller, and gas-handling module electronics see Figure 2-3.

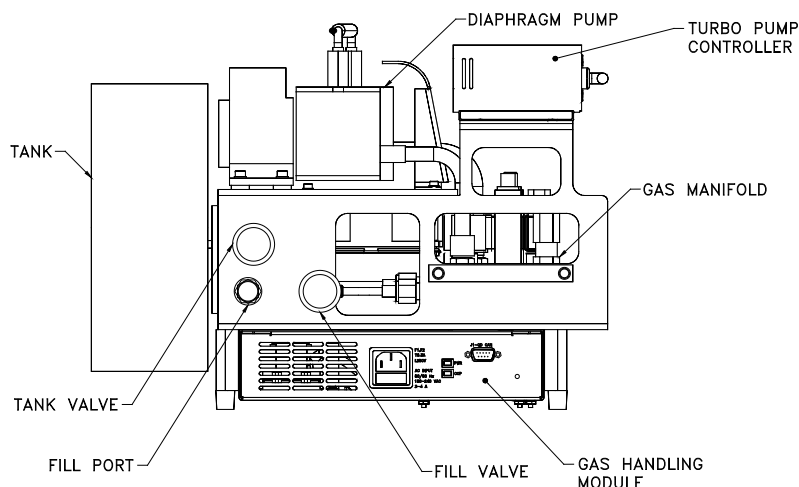


Figure 2-3. DR Gas-Handling System

2.4.1 Diaphragm Pump

The diaphragm pump maintains the outlet pressure from the turbo pump at a few torr. This is necessary for proper operation of the turbo pump. The diaphragm pump is hermetically sealed and uses no lubricants that might otherwise contaminate the gas mixture. During normal operation, the outlet of the diaphragm pump exhausts into the refrigerator insert to maintain circulation.

2.4.2 Mixture Tank

A 4-liter tank is permanently mounted on the side of the gas-handling module to store the ^3He - ^4He mixture. The tank is connected to the valve manifold via a stainless steel tube with a manual valve in line to seal the tank off. The manual tank valve, labeled TANK, is used to secure the helium mixture in the tank when maintenance is needed. During normal operation the manual valve should always remain open.



WARNING !

Closing the manual tank valve while operating the Dilution Refrigerator can cause the helium gas mixture to be lost from the system. Do not close the TANK valve unless instructed to do so by a maintenance wizard or Quantum Design personnel.

2.4.3 Gas Manifold

The gas manifold contains the valves, pressure gauges, and hose connections for the gas-handling system. There are 3 solenoid valves on the manifold; Supply, Bypass, and Tank Isolation. All of these valves are controlled by the gas-handling electronics and are open when not energized. The manifold also houses two pressure gauges that monitor the diaphragm pump outlet and the turbo pump foreline pressures. At the back of the manifold is a quick-disconnect fitting, used with the nitrogen cold trap for cryocleaning the gas mixture.

2.4.4 Turbo Pump Controller

The turbo pump controller is mounted on top of the gas handling system and provides the power and speed control for the turbo pump on the probe. The controller is connected to the turbo pump via a multi-conductor cable located inside the probe tether. Communication with the controller is handled by the gas-handling electronics through a serial cable. Operation of the turbo pump is fully automated.

2.4.5 Gas-Handling Module

The gas-handling module contains the electronics necessary to run all other components of the gas handling system. The module is located at the base of the gas handling system. The PC communicates with the module via the CAN bus connector. The entire gas handling system is powered through the module's power connector.

2.4.6 Cart and Step Stool

The Dilution Refrigerator system is incorporated into a wheeled cart for safe storage and sample mounting. Once the system is assembled, the gas-handling system is located inside the cart with the probe up front, resting on the turbo pump, as shown in the Figure 2-4 below. A latching mechanism, integrated into the cart, is used to secure the Dilution Refrigerator probe while on the cart. A hole in the back of the cart provides access to the quick-disconnect fitting used for cryocleaning the mixture. The cart can be rolled out of the way when the system is not needed.



Figure 2-4. DR Cart Assembly

A step stool is also provided with the Dilution Refrigerator system. It is strongly recommended that the stool be used during insertion and removal of the DR probe from the PPMS sample chamber. The shaft of the probe is extremely fragile, and the turbo pump is quite heavy by comparison. This combination makes the probe particularly susceptible to damage during handling. Although there is nothing special about the stool, it is included with the system to facilitate safe handling of the insert.

2.5 Model CM-D (Temperature Control Module)

The Model CM-D temperature control module (Figure 2-5) provides all of the necessary electronics to read and control temperature on the Dilution Refrigerator. The module has two heater drivers and two low-power resistance bridges. These are used to operate the still heater, still thermometer, sample heater, and sample thermometer. The module uses feedback control to maintain temperature set points received from the user or the control software.

At low temperatures, it is difficult to avoid self-heating when using resistive temperature sensors. This is especially true below 100 mK. The resistance bridges in the module are designed for low power dissipation when reading the temperature sensors so that self-heating is minimized.

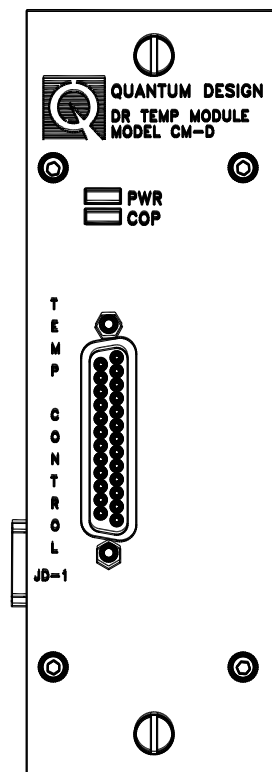


Figure 2-5. Model CM-D Temperature Control Module

2.6 Model 1000 (Modular Control System)

The Model 1000 modular control system is a general-purpose chassis (Figure 2-6) that houses, cools, and provides power to CAN-based electronics modules. The Model 1000 can accommodate up to six electronics modules for PPMS options. The Model CM-D temperature control module resides in the Model 1000. Each module plugs into the backplane which provides the module with power as well as a CAN network connection to the computer (PC). In addition, there is an extra CAN connection on the rear panel of the Model 1000 for connecting an external CAN controller if required by an option. The Dilution Refrigerator gas handling system connects to the CAN bus via this connector.

See the *Model 1000 Modular Control System User's Manual* for more information about the Model 1000.

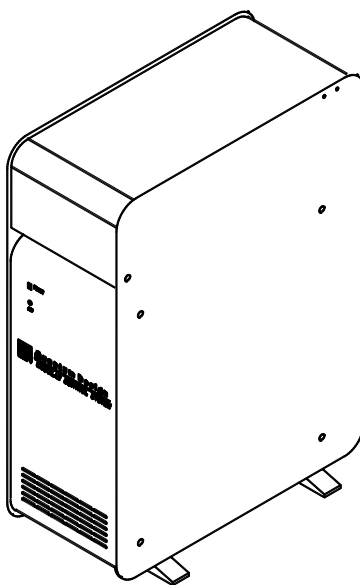


Figure 2-6. Model 1000 Modular Control System

2.7 CAN Computer Interface Kit

The CAN computer interface (or network adapter) kit contains the CAN-based network adapter, cable, and software that are needed to connect the Model 1000 to the computer (PC).

2.8 Electrical Connections and Cables

2.8.1 Gas Handling System CAN Cable

The PC software communicates with the gas handling system via the CAN bus. The gas handling system connects to the CAN bus at the back of the Model 1000 using the gas handling system CAN cable, part number 3091-401. The cable connects to the Model 1000 port labeled “J3-QD CAN” and to the gas handling module port labeled “J1-QD CAN.”

For systems with the Dilution Refrigerator, the CAN bus is terminated in the end of the gas handling system CAN cable. Therefore, the Model 1000 “TERMINATOR” switch should be set to “OUT” and the gas handling system CAN cable should always be left connected to the Model

1000. The cable can be disconnected from the gas handling module and the Dilution Refrigerator moved away from the PPMS when it is not in use.

2.8.2 Temperature Cable and RF Filter

The temperature cable is used to connect the Dilution Refrigerator probe to the dilution refrigerator Model CM-D temperature control module when the probe is inserted into the PPMS. This cable makes connections to the two heaters and two thermometers necessary to run the DR. The cable plugs into the upper 12-pin Fischer connector, labeled TEMPERATURE, on the right side of the probe head, and into the 25-pin “D”-shell connector (JD-1) on the Model CM-D temperature control module. A pinout table of the temperature cable can be found in Appendix B.

The metal box at the refrigerator insert end of the temperature cable contains an electrical filter to remove radio frequency (RF) interference from the leads that enter the dilution refrigerator insert. RF power can cause thermometer self-heating, especially below 100 mK, making this filtering necessary to achieve accurate temperature readings. The filters are designed to remove enough RF power for operation in typical laboratory environments. However, if you have excessive RF power in your laboratory, you may still experience thermometer self-heating.



Figure 2-7. Temperature control cable

2.8.3 Sample Measurement Cables and RF Filters

Sample connections are made with a measurement-option specific cable that plugs into option electronics and the 10-pin Fischer connector, labeled SAMPLE, on the right side of the probe head. Refer to the option manual for specific information regarding a particular measurement option. Pin-outs for DR heat capacity and DR user experiment cables are in Appendix B.

The sample measurement cable also has an RF filter to remove RF from the sample measurement leads. This filtering is necessary because some experiments may be susceptible to self-heating errors due to RF. Additionally, RF radiation from the sample leads could be received by the thermometer leads, causing thermometer self-heating. The RF filter on the sample measurement cables prevents this self-heating.

2.9 Liquid Nitrogen Cold-Trap Assembly

The Liquid Nitrogen Cold Trap removes contaminants from the ^3He - ^4He mixture. As described in Section 5.2.1, a wizard called, "Cryoclean Mixture Using LN2 Trap," leads you through the cryocleaning process.

2.9.1 Cold-Trap

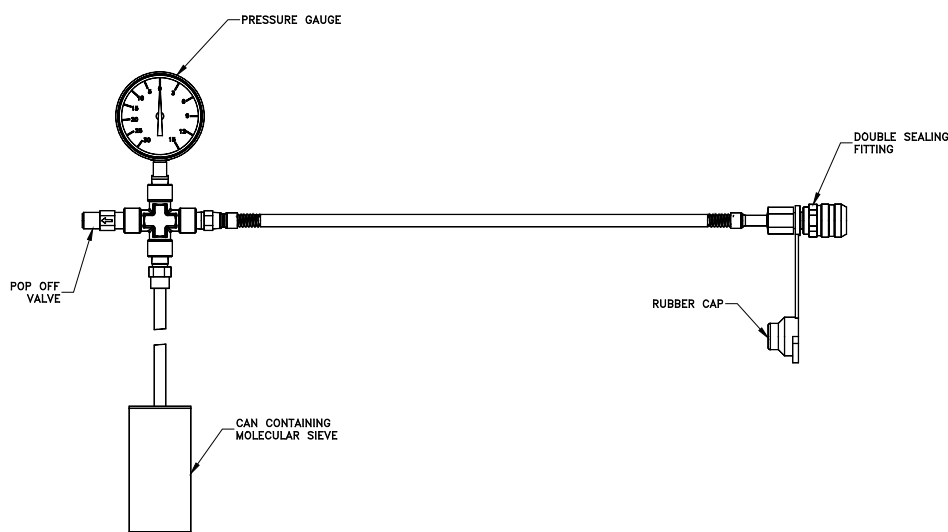


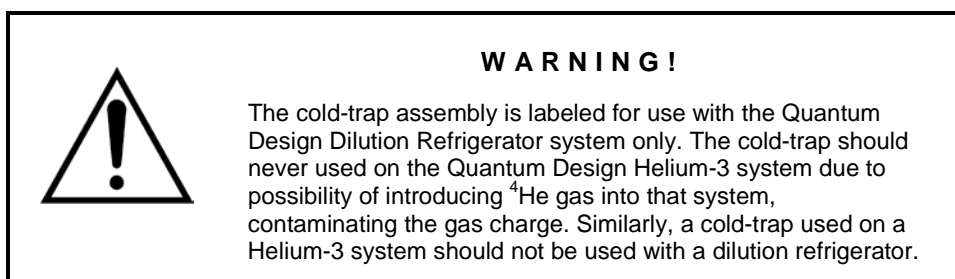
Figure 2-8. Liquid nitrogen cold-trap for DR

The cold-trap consists of a stainless steel can containing molecular sieve material, a gauge to monitor the pressure, a pop-off valve to prevent overpressure, and a quick-disconnect fitting to connect to the gas-handling manifold as shown in Figure 2-7. When cooled in liquid nitrogen and filled with helium mixture, the molecular sieve readily absorbs any non-helium gas or vapor, thereby removing contaminants from the mixture.

The pressure gauge indicates the quantity of gas in the assembly. Normally, when not connected to the gas-handling manifold, this gauge reads to the left of zero at room temperature, indicating a vacuum. However, after performing many cryoclean procedures, contaminant gas can build up in the trap. If the gauge reads to the right of zero at room temperature, the pressure inside the cold trap is greater than ambient pressure and the cold-trap needs to be pumped out. This procedure is

described in Section 5.2.2. The wizard called, “Regenerating LN2 Trap,” leads you through the regeneration process. When the cold-trap is at liquid nitrogen temperature and no helium gas has been introduced to the trap, the pressure gauge should read 30 in. Hg (to the left), indicating a hard vacuum. If the pressure gauge fails to reach 30 in. Hg vacuum, the trap may need to be regenerated or may have a leak.

The cold-trap connects to the gas-handling manifold using the double sealing quick-disconnect fitting. The corresponding fitting on the manifold is accessed through the hole at the back of the cart. These fittings completely seal when not connected, preventing atmospheric gases from entering either the refrigerator helium mixture or the cold-trap. Each time the cold-trap is connected to the gas-handling manifold a thin film of Apiezon M Grease should be applied to the mating surfaces of the quick-connect fitting in order to lubricate the O-rings in the fittings. Place the rubber dust-cap on both fittings when they are not in use to prevent dirt from accumulating.



2.9.2 Cold-Trap Regeneration Fitting

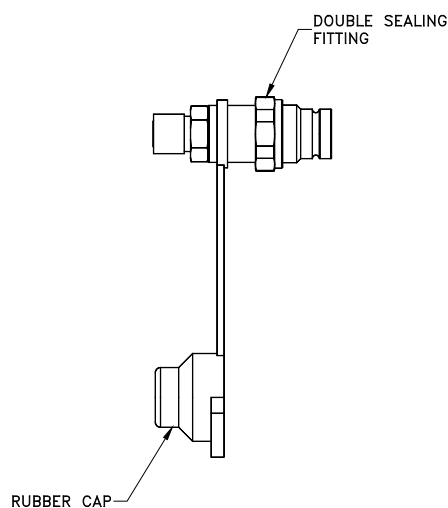


Figure 2-9. Cold-Trap Regeneration Fitting

The cold-trap regeneration fitting allows you to pump out the cold trap if the gas pressure gets above atmospheric pressure in the trap. The fitting adapts from the double sealing fitting on the cold trap assembly to the system pump-out hose, which is shown in Figure 5-1. The system pump-out hose then attaches to the top plate of the PPMS to allow pump-out of the cold trap using the PPMS high vacuum system. This procedure is described in more detail in

“Regenerating the Cold Trap” (Section 5.3.2). Place the rubber cap on the double sealing fitting when not in use to protect the fitting from dirt.

2.9.3 Liquid Nitrogen Dewar

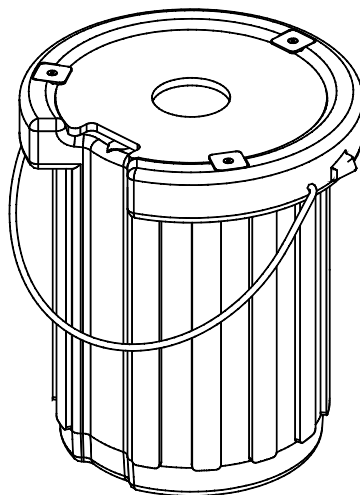


Figure 2-10. Liquid nitrogen dewar flask

2.10 Option Specific Hardware

2.10.1 Heat Capacity Measurements

If your system is equipped with the DR Heat Capacity Option, the following hardware is included to allow you to perform heat capacity measurements down to 50 mK.

Special Heat Capacity sample mounts have been developed for use with the Dilution Refrigerator system. These calorimeters have been designed to plug into the sample stage and the thermometer is optimized to run at temperatures down to 50 mK. Very thin wires are used to connect to the calorimeter to provide longer sample thermalization times than other calorimeters designed for higher temperatures. The wires are very delicate, so polymer strips are used for mechanical support of the calorimeter platform. An adapter is provided to allow the Heat Capacity sample mounts to be used with the standard sample-mounting station provided with the PPMS Heat Capacity system.

A special calibration fixture is included for performing the automated calibration procedure on a new Heat Capacity sample mount. Note that this calibration fixture is similar, but not identical, to one provided with Helium-3 systems for Helium-3 Heat Capacity Option. If you have both Helium-3 and DR Heat Capacity Option, you should be careful not to mix up either the fixtures or the sample mounts. Using the DR calibration fixture on the Helium-3 sample mounts could

results in damage to the sample mounts. Using the Helium-3 calibration fixture on the DR sample mount will result in a faulty calibration, because the fixture will not contact the heat capacity sample platform. Also, the copper contained in the Helium-3 calibration fixture and sample mounts causes excessively long relaxation times at high magnetic fields below 100 mK. You can distinguish the hardware for each heat capacity option by looking for a bare sanded surface. The DR option hardware has a bare silver surface where the gold plating is removed, whereas the Helium-3 option hardware has fully gold-plated copper surfaces. The bare silver surface on the DR option calibration fixture can be found on the top of the rectangular handle. The DR option sample mount has bare silver surfaces on the sides. See Figure 2-10 which illustrates these pieces of hardware.



Figure 2-11. DR Heat Capacity Sample Mount and Accessories

Heat capacity measurements on the Dilution Refrigerator are performed using the Model CM-E Heat Capacity Module (see Figure 2-11), which resides in the Model 1000. Heat capacity measurements on the Dilution Refrigerator cannot be performed using the older DSP card, which resides in the Model 6500. However, the Heat Capacity software is still used to make measurements using either the CM-E module or the Model 6500.

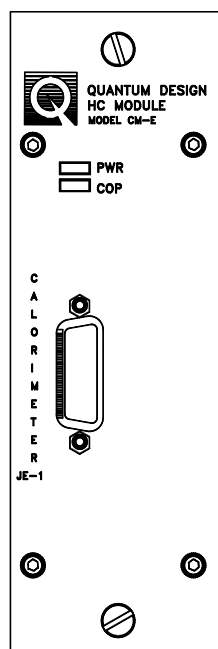


Figure 2-12. Model CM-E Heat Capacity Module

A cable is provided for performing heat capacity measurements on the Dilution Refrigerator (see Figure 2-12). This cable connects the model CM-E Heat Capacity Module to the DR probe, and provides RF filtering for the sample wiring that is used for heat capacity measurements.



Figure 2-13. DR heat capacity option cable

For more information about taking heat capacity measurements using the Dilution Refrigerator, refer to the *Physical Property Measurement System: Heat Capacity Option User's Manual*.

2.10.2 User Experiment Hardware

A User Experiment Kit is available to facilitate user-designed measurements. This kit includes three sample mounts and a sample cable with filter box.

The generic sample mounts have a flat surface where you mount your sample or experiment. Eight solder pads provide electrical connections for your experiment. Note that these sample mounts appear nearly identical to the Helium-3 resistivity sample mounts. However, the DR sample mounts are constructed from silver in order to avoid the large heat capacity of copper in high magnetic fields below 100 mK. You should not use Helium-3 sample mounts on the DR if you plan to apply a magnetic field above 1 T. You can distinguish the two sample mounts by looking at the straight edge of the metal plate. The DR sample mounts have a bare silver surface on one of these edges, whereas both edges of the Helium-3 sample mounts are gold-plated copper.

The user experiment cable connects to the “SAMPLE” Fischer connector and provides access to the eight experimental leads. These leads are terminated in a 25-pin “D”-shell connector, which you can plug into to adapt to your user experiment electronics. For a pinout of this cable, see Appendix B.



Figure 2-14. DR Sample cable

2.10.3 Electrical Transport Option (ETO) Measurements

A special adapter cable is required to use the Electrical Transport measurement option with the Dilution Refrigerator temperature option. The adapter cable (part number 3091-626) connects to both the ETO head and the DR probe sample wiring. This cable is supplied with the DR/ETO compatibility kit (8091-004) or with the DR option if the option was bought with ETO. Contact Quantum Design if you purchased the Electrical Transport option separately and required this cable. This cable pin out can be seen on Appendix B-5 of this manual.

ETO sample are mounted onto the DR probe using the DR transport sample mounts (4091-610) that ship with the DR. These mounts have a flat surface where you mount your sample or experiment. Eight solder pads provide electrical connections for your experiment. Note that these sample mounts appear nearly identical to the Helium-3 resistivity sample mounts (see Figure 2-9 of the Helium-3 User Manual). However, the DR sample mounts are constructed from silver in order to avoid the large heat capacity of copper in high magnetic fields below 100 mK. You should not use Helium-3 sample mounts on the DR if you plan to apply a magnetic field above 1 T. You can distinguish the two sample mounts by looking at the straight edge of the metal plate. The DR sample mounts have a bare silver surface on one of these edges, whereas both edges of the Helium-3 sample mounts are gold-plated copper.

Refer to the Electrical Transport Option User's Manual (1084-700) for information about wiring samples for ETO measurements to the eight-pad sample mounts and for information about taking ETO measurements when the DR insert is installed.

2.11 Other Accessories

- **Grease.** Apiezon N Grease and Apiezon M Grease are included with the Dilution Refrigerator. N grease is used on the contact fingers on the refrigerator insert for improved contact between the sample chamber and insert. N Grease can also be used between the sample mount and sample holder, as well as between the sample mount and sample. M grease is used to lubricate the O-ring on the refrigerator probe. Also, M grease is used on the cold trap quick-connect fitting as described in section 2.9.1
- **Sample Mounting Pins.** The sample mounting pins are used to immobilize the mixing chamber and sample stage whenever sample mounts are attached or removed from the sample stage. The mixing chamber support is somewhat delicate, so samples mounts should never be attached or removed without using the sample mounting pins. The sample mounting pins should not be used at any other time. In particular, the sample mounting pins must be removed before inserting the refrigerator probe into the PPMS. See Figure 2-13.



Figure 2-15. Sample mounting pins used to secure the mixing chamber.

- **Wrenches and screws.** A small hex ball driver is provided for mounting samples. Small screws are provided for attaching the sample mounts to the sample stage in case the original screws become worn. Additionally, two hex “L” wrenches are supplied for removal of the sample stage and adjustment of the pin guide tube. These procedures should be performed only under guidance from Quantum Design personnel.
- **Storage accessories.** When the probe is stored in the cart, the probe cover should be placed over the end of the probe in order to protect the delicate end. The vinyl O-ring cover should be placed over the O-ring in order to avoid accumulation of dust on the O-ring.

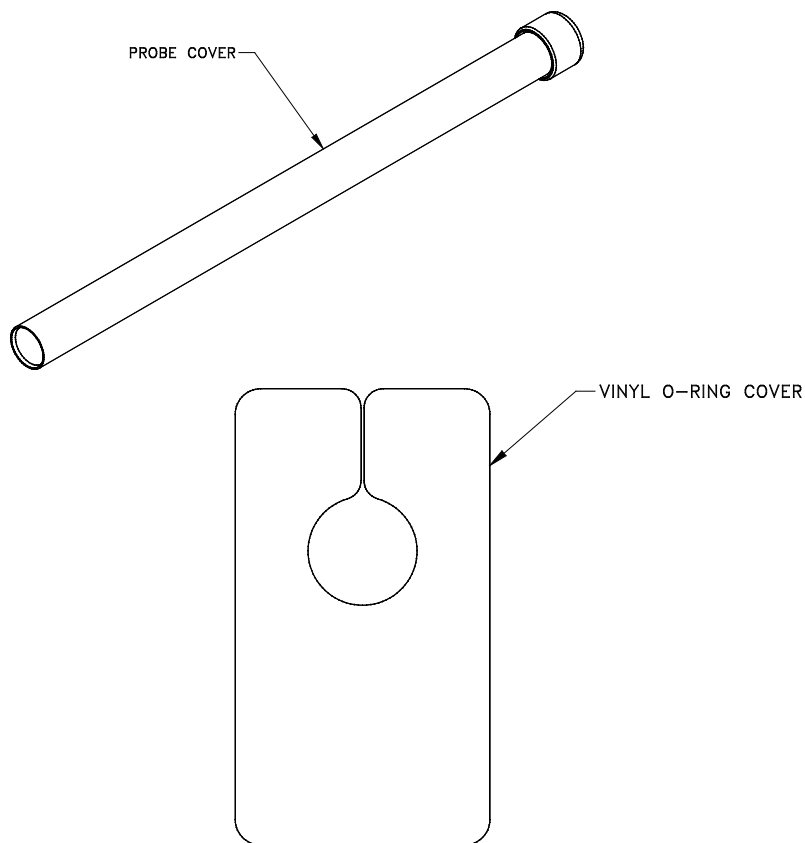


Figure 2-16. Storage Accessories

Software

3.1 Introduction

This chapter contains the following information:

- Section 3.2 discusses the architecture of the Dilution Refrigerator software.

3.2 Dilution Refrigerator Software Architecture

Dilution Refrigerator option software consists of a main temperature control state engine, a data logger, and automated maintenance wizards. All the necessary software is usually installed in the \qdppms\DROption directory. After installation, the dilution refrigerator software loads whenever MultiVu is started. The software detects and controls the dilution refrigerator automatically, implementing the various control modes described in Section 1.4. Only high-level state control is executed in the software. Real-time temperature feedback control is executed in the Model CM-D Temperature Control Module and the Model 6000 PPMS Controller. The maintenance wizards are included to perform routine operations required to maintain the Dilution Refrigerator system.

3.2.1 Temperature Command Redirection

The Dilution Refrigerator temperature control state engine is executed on the PC. However, new temperature set points are received by the 6000 in the usual manner. When an application or the user needs a new temperature set point, the set point is entered by sending a GPIB command to the Model 6000, executing a sequence with temperature commands, or by entering the new temperature set point directly from the front panel of the Model 6000. To ensure compatibility with existing software, a special software architecture has been chosen to preserve these interfaces while the main temperature control state engine executes on the PC.

When the Dilution Refrigerator software is initialized, which occurs when MultiVu starts, it sends a command to the Model 6000 that instructs it to forward future temperature commands to the control program executing on the PC. The Model 6000 then operates in this special redirected mode until temperature redirection is cancelled. Redirection is cancelled when the Dilution

Refrigerator probe is removed from the PPMS sample chamber or the Dilution Refrigerator software is shut down.

When redirection is active, temperature control status information is also redirected. The PC determines whether the temperature is “Chasing,” “Near,” or “Stable” and sets the status word on the Model 6000 appropriately. Any application programs, including PPMS MultiVu, determine the state of the temperature control engine by querying this status word.

Information appearing in the Model 6000 front panel indicates when the Dilution Refrigerator software is running and the commands are redirected. When temperature control is redirected, a small up-and-down arrow appears by the **TEMP** display. The arrow indicates that temperature commands are being redirected to the PC software. If you use the Model 6000 to enter a temperature, a “DR” indicator appears in the upper left corner of the Model 6000 front panel. Also, when you enter a temperature command from the Model 6000 or MultiVu, or a software application sends a temperature command via GPIB to the Model 6000, the stability indicator on the Model 6000 front panel displays “PC” until the software returns the status to the appropriate value. While “PC” status is indicated on the front panel, the PPMS temperature status word indicates an “unknown” temperature state.

In rare cases when the PC crashes or the software stops responding, a “PC” indicator may remain lit on the Model 6000 front panel and you do not see the normal status messages. To fix the problem, you must restart MultiVu, which will also restart the dilution refrigerator option software. The software will automatically recover from the crash. If you want to recover normal PPMS control without restarting the software, you can terminate the command redirection on the Model 6000 by entering the **CTRL** menu, selecting item 4, and then entering 2.

3.2.2 Dilution Refrigerator Temperature Reporting

In the normal PPMS architecture, MultiVu software and measurement option software read the sample temperature from the Model 6000. However, the Model 6000 normally reports the PPMS block temperature, which is not the sample temperature when the DR is installed. With the Dilution Refrigerator, the Model CM-D Temperature Control Module measures the Dilution Refrigerator sample temperature. In order to preserve the PPMS architecture, the sample temperature is sent to the Model 6000 by the Dilution Refrigerator software and reported on the Model 6000 front panel. PPMS MultiVu and measurement option software then read the sample temperature from the Model 6000 in the usual way.

3.2.3 Software Initialization

After the Dilution Refrigerator option software has been installed, the software will load automatically when MultiVu is started. Normally, the software is initialized with the DR disconnected or not inserted in the PPMS. In that case, the software runs in the background and does not affect normal operation of the PPMS.

Depending on the condition of the system, the software will enter one of the following states after initialization:

- **DR Not Connected.** If the CAN cable or line power is not connected to the Dilution Refrigerator gas handling system, then the software enters the “DR Not Connected” state. In this state, the Dilution Refrigerator software runs in the background and does not affect normal operation of the PPMS.

- **Probe Out.** If the gas handling system is detected (CAN cable and power connected), but the Dilution Refrigerator probe is not inserted into the PPMS sample chamber, the software enters the “Probe Out” state. In this state, the Option Control Console will be displayed and a message will indicate that the Dilution Refrigerator has been detected. The temperature control aspect of the software runs in the background and does not affect normal operation of the PPMS.
- **DR Standby.** If the probe is inserted into the PPMS and the gas handling system is detected, the system enters the “DR Standby” state, provided the PPMS is near room temperature. Temperature redirection is active, and the system will be initialized for temperature control when a temperature set point is entered.
- **Controlling.** If the probe is inserted into the PPMS, the gas handling system is detected, and the PPMS is cold, the system begins controlling temperature. The system initializes temperature control automatically. Note that this condition should occur only if the software is exited and then restarted while the probe is inserted into the PPMS, a rare condition.

3.2.4 System Configuration and Calibration

After the DR probe is inserted into the PPMS and a new temperature set point is received, the software initializes the hardware and performs a self-test. The hardware initialization performs various operations to ensure that the hardware is ready to control the DR. During this time, configuration and calibration information is read from a serial ROM in the gas handling module. Included in this information is the calibration for the DR sample thermometer. No system-specific configuration or calibration information is stored on the PC. As a result, you can use the DR on any PPMS that is equipped with the necessary hardware without moving any files. Also, you can use multiple different Dilution Refrigerators on one PPMS without changing any files.

3.2.5 DR Option Control Console

The DR Option Control Console window (see Figure 3-1) is used to access status information about the Dilution Refrigerator and control system data logging. You can also shut down the temperature control engine when necessary. The **DR Option Control Console** is accessible in the MultiVu software under the **Instrument>>Dilution Refrigerator** menu.

The **Status** panel provides a short description of the current state of the DR temperature control state engine.

The **Logging** button opens the **Log DR Data** window from which you can monitor and log different hardware parameters related to the option. This procedure is discussed in detail below.

The **Shut Down DR** button prompts the DR option to enter the standby state. In the standby state, the DR temperature control software is idle while it waits for a new set point, and the DR hardware is shut down.

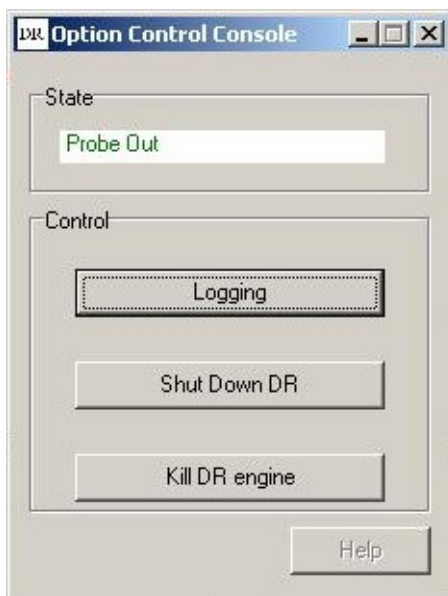


Figure 3-1. DR Option Control Console

3.2.5.1 DATA LOGGING

It may be necessary to monitor the various state variables of the DR temperature control engine. This is usually required only to diagnose problems with the Dilution Refrigerator. Logging is accomplished using the **Log DR Data** dialog. The **Log DR Data** dialog can be accessed by pressing the **Logging** button on the **DR Option Control Console** or through the menu item **Utilities>DR>Log DR Data**. The data logger produces files that are compatible with the PPMS MultiVu data file format and can therefore be displayed in real time within PPMS MultiVu.

To use the logger, (1) select the file to which the diagnostic data will be written, (2) select a logging frequency, (3) choose the fields to log, and then (4) push the Start button. The logger is configured to begin logging whenever the DR probe is inserted into the PPMS and will append data to an existing file by default. The logger will only allow the Overwrite Existing File check box to be checked when logging is stopped.

The **Message Log** tab lists messages or alarms posted by the DR temperature control software. This tab is used for diagnostic purposes only.

3.2.5.1.1 Logging Set Up

The **General** tab in the **Log DR Data** window allows you to set up the log file by either browsing over existing files or by entering a new file name in the **Log File Name** text box. Note that you cannot select a new data file if logging is started. You must first press the **Stop** button to stop logging. The logging interval and the maximum file size are also set here. The default maximum file size is set to 20 MB and the interval to 20 seconds. When the log file reaches the maximum file size, the first half of the data file is purged, leaving only the most recent data in a file of half the maximum size. Pressing the **View** button automatically opens a graph in MultiVu of the current log file. The **Start** and **Stop** buttons are used to control logging activity.



Figure 3-2. General Tab in Log DR data

3.2.5.1.2 Selecting the Fields

The data being logged to file can be selected from the **Temperature**, **Gas Handling**, and **Diagnostic** tabs on the **Log DR Data** dialog by marking the check boxes to the left of the desired fields. Also shown in the tabs are the real-time values of each field displayed in the text box to the right. A description of each data fields is given below.

Temperature Tab

Temperature Status	A numerical status code indicating the current temperature status of the PPMS block, DR sample, and DR still. This can be decoded using the DR status calculator.
Block Temperature	The temperature reported by the PPMS sample chamber block.
Sample Temperature	The temperature reported by the DR sample stage thermometer, located near the sample.
Still Temperature	The temperature reported by the still thermometer, located on the still pot on the DR probe.
Sample Setpoint	The last temperature setpoint sent to the temperature control module for the sample. This is the setpoint used by the temperature control module when actively controlling temperature. This temperature may not coincide with the DR Setpoint depending on the state of the temperature control software.
DR Setpoint	The temperature setpoint for the Dilution Refrigerator system.
Sample Heater Current	The approximate current being applied to the sample heater, located near the sample.
Still Heater Current	The approximate current being applied to the still heater, located on the still pot on the DR probe.

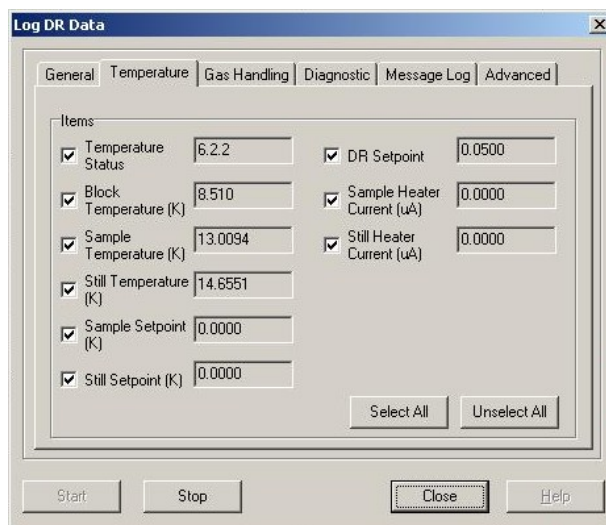


Figure 3-3. Temperature tab of Log DR data

Gas Handling Tab

Bypass Valve	The state of the Bypass valve. "1" indicates that the valve is open.
Supply Valve	The state of the Supply valve. "1" indicates that the valve is open.
Tank Isolation Valve	The state of the Tank Isolation valve. "1" indicates that the valve is open.
Outlet Pressure	The pressure, in Torr, as reported by the gauge connected to the outlet of the diaphragm pump.
Foreline Pressure	The pressure, in Torr, as reported by the gauge connected to the foreline of the turbo pump (inlet of the diaphragm pump).
Diaphragm Pump Speed	The diaphragm pump speed. Zero is stopped and 100% is full speed.
Turbo Pump Speed	The turbo pump speed as reported by the turbo pump controller, generally between 15 and 60 kRPM.
Turbo Pump Current	The turbo pump drive current as reported by the turbo pump controller.
Turbo Pump Temperature	The temperature of the turbo pump as reported by the turbo pump controller. The controller will shut down the pump if this temperature exceeds 60 °C.
Turbo Pump Status	The status of the turbo pump as reported by the turbo pump controller. (list states, and numerical values when plotting them)

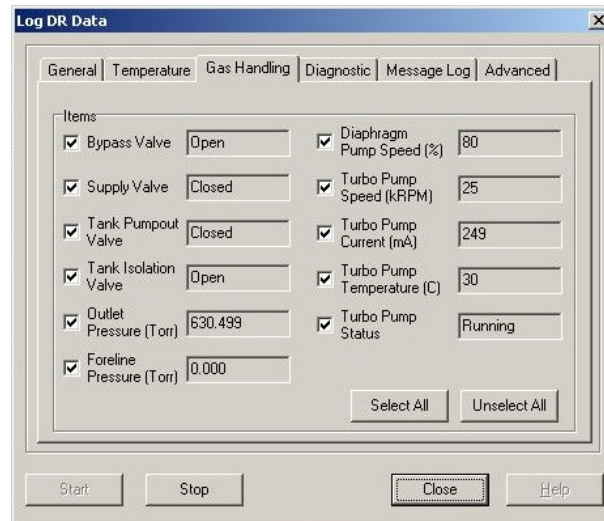


Figure 3-4. Gas Handling tab of Log DR data

Diagnostic Tab

Sample Thermometer Resistance	The resistance of the sample thermometer.
Sample Thermometer Current Excitation	The current being applied to the sample thermometer.
Still Thermometer Resistance	The resistance of the still thermometer.
Still Thermometer Current Excitation	The current being applied to the still thermometer.
Probe Switch State	The state of the switch on the DR probe which indicates whether or not the probe is inserted in the PPMS.
Magnetic Field	The magnetic field at the sample, as reported by the Model 6000.
Chamber Pressure	The pressure in the PPMS sample chamber, as reported by the Model 6000.
State ID	ID number corresponding to the current state of the state engine. This can be decoded using the DR status calculator.
Event	Event, encoded as a number, indicating the most recent event processed by the state engine. List event types and numerical codes.

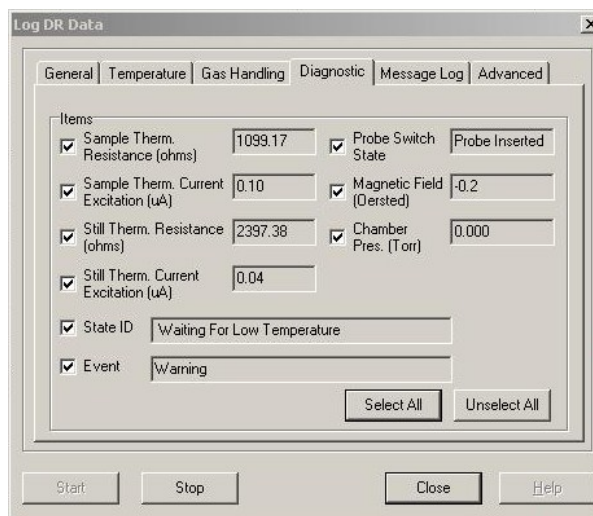


Figure 3-5. Diagnostic tab of Log DR data

3.2.5.2 SHUTTING DOWN THE DR TEMPERATURE ENGINE

The DR temperature control software can be shut down using the **Shut Down DR** button on the **DR Option Control Console**. This will end active temperature control by the software and place the software in the **Standby** state. In standby both the turbo pump and diaphragm pump are turned off and all valves are opened. The temperature control module will turn off all heaters but continue to report temperatures. The software will remain in the **Standby** state until another temperature set point is received.

Warning: Pressing the **Shut Down DR** button during normal operation can significantly warm the Dilution Refrigerator and cause large delays to reestablish temperature stability.

3.2.6 Status Calculator

The temperature status and engine state are both recorded by the DR data logger. If you wish to decode these items into human-readable form, use the **DR Status Calculator**, which is accessed through the menu item **Utilities>DR>Status Calculator**. To read and decode the current temperature status and engine state, push the **Current Status** or **Current State** button. To decode a value recorded to a file, manually enter a state or status code, then push the **Decode Status** or **Decode State** button.

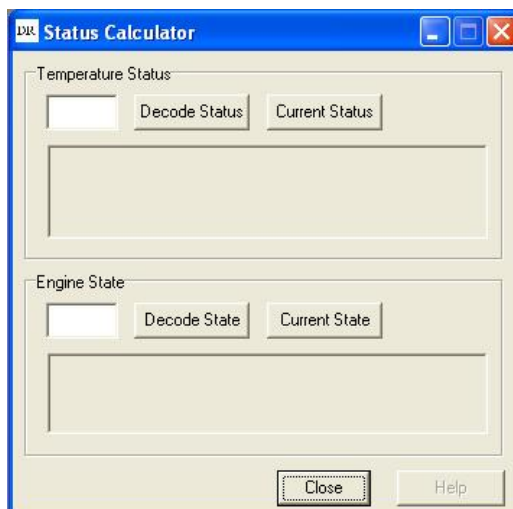


Figure 3-6. DR status calculator

3.2.7 DR Option Wizards

A number of software wizards are provided to perform a variety of maintenance and diagnostic procedures. To run a wizard, select the MultiVu menu option **Utilities>>DR>>Wizards**. It is necessary to put the DR in the standby state before running a wizard. While a wizard is running, the temperature control software automatically suspends active control and “Wizard Running” is displayed in the status window of the **DR Option Control Console**. When the wizard is finished, the temperature control software returns to the standby state, awaiting a new temperature setpoint.

The table below lists the available wizards and gives a short description of the function. Each Wizard is discussed in more detail in Section 5.2.

Check DR Impedance Flow at 20K	Checks the impedance flow rate at 20 K with the probe inserted into the PPMS.
Cryoclean Mixture Using LN2 Trap	Cleans the mixture using the cold-trap cooled in the LN ₂ filled dewar. The wizard allows you to choose the number of cleaning cycles performed.
Evacuate and Seal	Evacuates the Dilution Refrigerator system after it has been open to atmosphere for maintenance reasons.
Regenerate LN2 Trap	Pumps contaminant gases out of the LN ₂ cold-trap using the PPMS sample chamber high vacuum.
Secure Gas Mixture	Secures the helium gas mixture in the tank on the gas-handling system.
Regenerate DR Sorb	Regenerates the charcoal sorb located on the DR condenser using the PPMS sample chamber high vacuum. This Wizard needs to be run every 15 thermal cycles to ensure proper performance.

System Operation

4.1 Introduction

This chapter contains the following information:

- Section 4.2 explains how to handle the Dilution Refrigerator probe.
- Section 4.3 explains how to set up and shut down the Dilution Refrigerator.
- Section 4.4 summarizes procedures for performing measurements with the DR.
- Section 4.5 explains the disconnecting and storing the hardware.

4.2 Handling the DR Probe

The Dilution Refrigerator probe is delicate and should be handled with care. It should always be supported by the turbo pump at the head of the probe as shown in Figure 2-1. Hold the main shaft of the probe only to carefully guide it. The tubing used to fabricate the main shaft of the probe is very delicate and will not support the full weight of the turbo pump and tether. When inserting the probe into the PPMS sample chamber it is very important to avoid stressing the shaft. Keep the probe well aligned with the chamber. Large misalignments will bend the shaft or damage the contact fingers. The step stool provided with the system should always be used when inserting or removing the probe to help the user properly align the probe.

When the DR probe is on the cart it rests on the turbo pump with the safety latch down to secure the probe. The DR probe should always be secured on the cart in this manner when it is not inserted into the PPMS sample chamber. Also, the probe cover should be placed over the end of the probe in order to protect it while it is on the cart.

4.3 System Setup

Note: If you are installing the Dilution Refrigerator system for the first time, please refer to the detailed installation instructions in Appendix A.

After the Dilution Refrigerator has been set up for the first time, very little work is required to shut down and reactivate the system between each use. The DR software is running whenever MultiVu is open, and automatically detects the DR when the gas handling module is powered up and the CAN bus cable is plugged in.

The following instructions assume that the DR has been previously disconnected from the CAN bus for storage. The instructions begin with reconnecting the system. However, you may choose not to disconnect the system between usages if sufficient space is available for storing the DR cart adjacent to the PPMS.

4.3.1 Connecting the Electrical Cables

1. Position the DR cart close to the PPMS and Model 1000 such that the DR probe can easily be inserted into the PPMS.
2. Connect the CAN cable from the rear panel of the Model 1000 to the connector labeled “J1-QD CAN” on the DR gas handling system
3. Plug the power cable into the DR gas handling system. To reduce the possibility of ground loops the gas handling system should be plugged into the same power strip as the PC and Model 1000
4. Make sure the CAN cable from the PC to the Model 1000 is plugged in and the Model 1000 is powered.
5. Connect the temperature cable to the port labeled “JD1-Temperature” on the Model CM-D Temperature Control Module, located inside the Model 1000.

4.3.2 Cryoclean the Helium Mixture

If the Dilution Refrigerator gas mixture has not been cryocleaned within the last week, you should perform a cryoclean operation before attempting to use the system. Refer to Section 5.2.1 for details about this procedure.

4.4 Performing Measurements with the Dilution Refrigerator

The following procedures assume the Dilution Refrigerator system has already been set up (section 4.3). The DR probe should be removed from the PPMS sample chamber and secured onto the cart. For information about using a specific PPMS measurement option with the DR, please refer to the appropriate option manual.

4.4.1 Mounting and Un-Mounting Samples

1. Follow the sample preparation and mounting instructions to mount a sample onto the sample mount. These instructions are contained in the specific user manual for the measurement option being used.
2. Unscrew and remove the radiation shield from the end of the DR probe. Note: Hold onto the condenser tube to prevent it from unscrewing as well. See Figure 4-1.



Figure 4-1. The radiation shield unscrews from the condenser tube.

3. Insert the sample mounting pins into the condenser tube to stabilize the dilution unit. Be careful to watch that the pins mate with the mixing chamber so that they do not push excessively on the mixing chamber, possibly causing damage to the dilution unit. The sample stage should move no more than a few millimeters when inserting the pins. See Figure 4-2.



Figure 4-2. Install sample mounting pins prior to mounting or dismounting samples.

CAUTION!

Never attempt to mount or dismount samples from the Dilution Refrigerator without first inserting the sample pins. Doing so could cause damage to the dilution unit.

4. Apply a small amount of Apiezon N Grease to the bottom side of the sample mount. Applying additional grease may be unnecessary if grease is left over from a previous run.
5. Plug the sample mount into the sample stage at the end of the DR probe. Be sure to orient the guide hole on the sample mount to align it with the guide pin on the sample stage.
6. Use the 0.050 inch hex ball driver supplied with the system to gently tighten the two hex screws on the bottom of the sample stage plate. Be careful to hold the ball driver as straight as possible so that you do not strip the screw heads. Do not over tighten the hex screws. Tighten the screws just enough to hold the plug-in sample mount against the sample stage. See Figure 4-3.



Figure 4-3. Tighten hex screws on sample stage.

7. Remove the sample mounting pins from the condenser tube.
8. Screw the radiation shield back onto the condenser tube. The radiation shield should only be snug.
9. Unscrew the top of the radiation shield and examine the sample stage and sample mount. Make sure there are no touches between the sample stage and the radiation shield. Even a small touch will prevent the Dilution Refrigerator system from reaching base temperature. See Figure 4-4.



Figure 4-4. Ensure there are no touches between the sample stage and radiation shield.

10. If no touch is found screw the top of the radiation shield back into place.

4.4.2 Inserting the DR Probe

CAUTION!

Handle the DR probe very carefully. To help prevent the probe from being damaged, use the provided step stool when you insert or remove the probe from the PPMS sample chamber. Support the weight of the probe using the turbo pump, not the delicate shaft of the probe. Handle the shaft of the probe only to guide it into the sample chamber.

1. Start the PPMS MultiVu software if it is not already running.
2. Apply a small amount of Apiezon N Grease to the contact fingers on the condenser tube and the probe shaft. The grease should not be visible but the contact fingers should feel tacky.
3. Inspect the soft silicone O-ring at the turbo pump base. Make sure it is clean and well greased. It may be necessary to clean it and apply a small amount of Apiezon M Grease to ensure a proper seal.
4. Place the step stool next to the PPMS dewar.
5. Start up the installation wizard that will step you through the procedure to run a sample measurement for your measurement option. The installation wizard sets the PPMS to room temperature and floods the sample chamber with helium. If you are not using a PPMS measurement option, vent the sample chamber using MultiVu by selecting the menu item **Instrument>Chamber** and then press the **Vent Cont.** button.
6. Remove the clamp around the sample chamber access port. Remove the blank flange from the PPMS access port. Check that the sample chamber is empty. If a puck is mounted into the PPMS it must be removed prior to inserting the DR.
7. Open the safety latch on the Dilution Refrigerator cart and remove the DR probe from the cart. Be sure to hold the DR probe at the turbo pump base. Carefully rotate the DR probe such that the tether is not twisted and the turbo pump is on top.
8. Step onto the step stool and raise the DR probe over the PPMS access port. The black mesh tether and two Fischer connectors on the probe head should be pointing to the right.
9. Slowly insert the DR probe into the PPMS sample chamber keeping the probe well aligned with the chamber. The resistance due to the contact fingers should increase over the last 5-6 cm.
10. Make sure the O-ring surface is properly seated onto the PPMS access port flange.
11. Plug in the temperature cable from the Model CM-D Temperature Control Module into the upper Fischer connector, labeled "TEMPERATURE", on the right side of the probe head. Plug the sample cable for your measurement option into the lower Fischer connector, labeled "SAMPLE".

Complete any additional tasks the installation wizard prompts you to perform before you run the measurement. This will include purging the sample chamber. If you are not using a PPMS measurement option, purge the sample chamber. In PPMS MultiVu, select the menu item **Instrument>Chamber** and then press the **Purge/Seal** button.

Note: Do not attempt to cool down the Dilution Refrigerator with the sample chamber in any state other than purged. Doing so could result in a failed cool down.

4.4.3 Running the Measurement

Run the measurement as you would normally. All procedures for running measurements in immediate mode or sequence mode are identical whether or not the Dilution Refrigerator probe is installed. However, when the probe is installed you can take measurements to 50 mK.

4.4.4 Removing the Dilution Refrigerator Probe

1. Set a temperature between 295 K and 300 K. When the sample chamber reaches room temperature the software will enter the Pausing to Equilibrate state. The software will remain in this state for 30 minutes to allow the entire probe to warm. The software will then enter the DR Warmed Up state. All valves will open and the pumps will shut off.

Note: Waiting for less than 30 minutes between 295 K and 300 K may result in a cold dilution unit and condenser when you remove the probe from the PPMS. The upper parts of the sample chamber remain cold for extended periods after warming the system, and the condenser may be cooled as it is pulled through the chamber during probe removal. Water may condense on the dilution unit, saturating the charcoal sorb.

2. In MultiVu, under the **Instrument** menu option, open the **Chamber** dialog box. Select **Vent Cont.** to vent the PPMS sample chamber.

CAUTION!

Avoid stressing the shaft of the refrigerator probe in any way while you are removing the probe from the sample chamber. The material out of which the shaft is constructed is thin and can easily bend or dent.

3. Step onto the step stool and carefully pull the DR probe straight up and out of the PPMS sample chamber. Support the probe by the turbo pump only.
4. Rotate the probe such that the tether does not twist then set it on the cart with the probe resting on the turbo pump. Close the safety latch on the cart to secure the probe. Place the probe cover over the end of the probe for protection.
5. Return the blank flange to the PPMS access port and clamp. Return to the **Chamber** dialog box in MultiVu and select **Purge/Seal** to pump out and seal the PPMS sample chamber.

4.4.5 Remove the Sample Mount

1. Unscrew and remove the radiation shield from the end of the DR probe. Hold onto the condenser tube to prevent it from unscrewing as well.
2. Insert the sample mounting pins into the condenser tube to stabilize the dilution unit. Be careful to watch that the pins mate with the mixing chamber so that they do not push excessively on the mixing chamber, possibly causing damage to the dilution unit. The sample stage should move no more than a few millimeters when inserting the pins

CAUTION!

Never attempt to mount or dismount samples from the Dilution Refrigerator without first inserting the sample pins. Doing so could cause damage to the dilution unit.

3. Use the 0.050 inch hex ball driver supplied with the system to loosen—but not remove—the two hex screws on the bottom of the sample stage plate. Be careful to hold the ball driver as straight as possible so that you do not strip the screw threads.
4. Remove the sample mount by pulling it straight up. The pins can easily be bent, so be careful.

4.5 Disconnecting and Storing the Hardware

Once the DR probe is back on the cart the Dilution Refrigerator system can be disconnected from the PPMS by unplugging the power cord and CAN bus cable from the gas handling system. The cart can then be wheeled away to a safe storage area. It is not necessary for the helium mixture to be secured during storage. However, if you plan to store the options for a period more than two months, perform the "**securing the Helium mixture**" wizard as seen in section 5.2.5. The Model 1000 can remain plugged in and powered if it is needed for other PPMS options. The gas handling system CAN cable should remain plugged into the Model 1000 because the CAN bus is terminated in this cable for systems with the Dilution Refrigerator.

CAUTION!

Failure to secure the gas before a long term storage may result in loss of "Helium mixture" gas.

Measurements

5.1 Introduction

This chapter contains the following information:

- Section 5.2 discusses the Dilution Refrigerator's maintenance procedures and wizards.

5.2 Maintenance Procedures and Wizards

All but one of the maintenance procedures described in this section are partially automated by using software wizards included with the Dilution Refrigerator option software. Each wizard discussed in the sections below consists of a series of automated operations and user instructions for completing a maintenance procedure.

To run a maintenance wizard, the Dilution Refrigerator temperature control must be shut down. If the DR probe is on the cart, then the temperature control is already shut down. If the probe is in the PPMS, you must press the **Shut Down** button on the **DR Option Control Console** before attempting to run a wizard. If you do not shut down the temperature control, the software will prompt you to shut down the control before running a wizard.



WARNING!

Pressing the **Shut Down DR** button during normal operation can significantly warm the Dilution Refrigerator and cause large delays while temperature stability is reestablished.

To run a maintenance wizard, select the MultiVu menu item **Utilities>DR>Wizards** and then select the appropriate wizard from the menu.

Some of the procedures require the use of the system pump-out hose (part number 4092-630) provided with the system. See Figure 5-1. This hose connects between the PPMS sample chamber and the Dilution Refrigerator gas handling system and uses the PPMS as a high-vacuum pumping station.

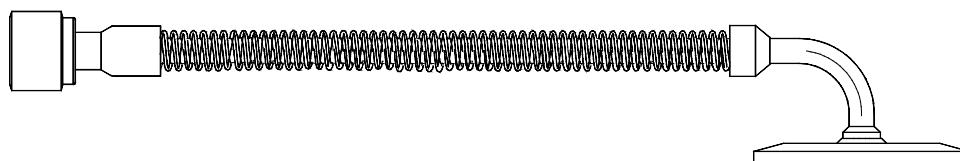


Figure 5-1. System Pump-out Hose (4092-630)

5.2.1 Cryocleaning the Helium Mixture Using LN₂ Cold Trap

Cryocleaning the helium mixture is necessary because atmospheric contaminant gasses build up in the mixture. These gasses freeze inside the DR probe, plugging the impedance and/or reducing condenser performance. To prevent these conditions, perform the cryocleaning procedure about once every week when the system is use, or if you have not used the system in more than a week. Very little or no helium mixture is lost during this procedure, so the operation can be performed often without the expense of adding helium mixture to the system.

To perform this procedure, you need the LN₂ cold trap (Figure 2-7), the small liquid nitrogen dewar (Figure 2-9), and liquid nitrogen to fill the dewar.



WARNING !

The cold-trap assembly is labeled for use with the Quantum Design Dilution Refrigerator system only. Use of this cold-trap on any other system could contaminate the mixture. The cold-trap should never be used on the Quantum Design Helium-3 system due to the possibility of ⁴He contamination. Similarly, a cold-trap used on a Helium-3 system should not be used with a dilution refrigerator.

To perform the operation, run the “Cryoclean Mixture Using LN₂ Trap” wizard. The software guides you through the following steps:

1. You are prompted to enter the number of cryoclean cycles to perform. The default value, 100, should normally be used. If it has been less than a week since you last cryocleaned the system, you may choose fewer cycles in order to shorten the time of the cryoclean operation. If you have not used the system for a long time, more than a month, then you may want to choose more than 100 cycles.
2. Wait while the wizard measures the amount of helium mixture in the system before the cryoclean.
3. Check to make sure that the LN₂ trap is not in an overpressure condition, in which case the trap should be regenerated using the “Regenerate LN₂ Trap” wizard.
4. Cool the trap by submerging it in liquid nitrogen. The trap is cold when bubbling of the LN₂ bath decreases.
5. Apply a small amount of Apiezon M grease to the double sealing connector on the cold trap, and then connect it to the DR gas handling system.
6. Wait while the wizard flushes the helium mixture in and out of the cold trap, removing all non-helium gasses.

7. Wait while the wizard pumps the helium mixture out of the trap.
8. Disconnect the cold trap from the gas handling system.
9. Wait while the wizard measures the amount of helium mixture in the system after the cryoclean. There should be the same amount of mixture before and after the cryoclean. If not, the wizard warns you that mixture may have been left in the trap, or air may have been introduced into the system.

The entire operation will take about 75 minutes to complete 100 cycles. If you choose a different number of cycles, the time may be shorter or longer.

5.2.2 Regenerating the LN₂ Cold Trap

If the room temperature pressure in the cold trap is greater than ambient (atmospheric) pressure, regenerate the cold trap by pumping the gas out of the cold trap. The pressure gauge on the cold trap reads zero when the pressure in the trap is at ambient pressure and reads to the right of zero when the pressure is greater than ambient. A reading to the left of zero indicates a vacuum condition and 30 inches Hg indicates a hard vacuum.

To regenerate the trap, run the “Regenerate LN2 Trap” wizard. The software guides you through the following steps.

1. Connect the cold trap to the PPMS sample chamber using the trap regeneration fitting and the system pump-out hose.
2. Wait while the wizard pumps out the cold trap using the PPMS high vacuum system.
3. Disconnect the cold trap from the trap regeneration fitting using the double sealing connectors, preserving the vacuum in the trap.

5.2.3 Regenerating the Charcoal Sorb

The charcoal sorb on the DR condenser may become partially contaminated with water when the DR probe is removed from the PPMS sample chamber. The upper region of the sample chamber remains cold long after warming the system. The DR condenser, where the sorb is mounted, can be cool enough to condense water when the DR is removed from the sample chamber. Therefore, you should regenerate the DR sorb once every 15 cool-down cycles in order to avoid problems with high vacuum that occur when the sorb is contaminated. The sorb is regenerated by pumping high vacuum on the DR probe while it is inserted into the PPMS sample chamber at room temperature.

To regenerate the sorb, run the “Regenerate Charcoal Sorb” wizard. The software guides you through the following steps:

1. Insert the DR probe in the sample chamber or set the sample chamber to room temperature if the probe is already inserted.
2. Wait while the wizard purges the sample chamber and then pumps high vacuum in order to regenerate the sorb. The wizard will wait for 30 minutes to pump out the sorb. You may choose to leave the system to pump longer if desired. If the sorb is very contaminated, the PPMS may signal a high vacuum error. The regeneration operation will still be successful if this occurs.

3. The wizard vents the sample chamber so that you may remove the DR probe. You may also purge the sample chamber and cool down the DR if desired.

5.2.4 Evacuating and Sealing the System

After the system has been opened for maintenance, it must be evacuated and the gas mixture stored in the tank must be let back into the system. Opening the system for maintenance is rarely required and should be done only under the guidance of Quantum Design personnel.

Before starting this procedure, verify that the system is in the helium-stored configuration with both the “TANK” and “FILL” valves closed. That is, you must have already run the “Secure Gas Mixture” wizard described in section 5.2.5. In rare cases you may need to perform the “Evacuate and Seal” procedure with the “TANK” valve open in order to pump old mixture out of the system. You should perform this procedure with the “TANK” valve open only if you plan to recharge the mixture using an external mixture tank supplied by Quantum Design.

To perform this operation, run the “Evacuate and Seal” wizard. The software guides you through the following steps:

1. Connect the FILL port on the DR gas handling system to the PPMS sample chamber using the system pump-out hose.
2. Wait while the wizard purges the system and then pumps high vacuum in order to evacuate the system.
3. Close the “FILL” valve.
4. Open the “TANK” valve to let the gas mixture back into the system.
5. Wait while the wizard measures the gas quantity recovered from the tank.

5.2.5 Securing the Helium Mixture

In order to save as much of the helium mixture as possible before performing any maintenance operation that requires opening the system, you must secure the helium mixture in the tank. Opening the system for maintenance is rarely required and should be done only under the guidance of Quantum Design personnel.

Before starting this procedure, verify that the system is in the normal operation configuration with the manual “TANK” valve open and the manual “FILL” valve closed.

To save the helium mixture in the tank, run the “Secure Gas Mixture” wizard. The software guides you through the following steps.

1. If the DR is inserted into the PPMS sample chamber and the temperature is less than 10 K, wait while the wizard warms the PPMS to 10 K.
2. Wait while the wizard pumps the helium mixture into the tank.
3. Close the manual “TANK” valve when instructed to do so by the wizard.
4. Wait while the wizard checks for a leak in the manual “TANK” valve, which could cause a loss of mixture when you open the system.

5.2.6 Checking the DR Impedance Flow

When troubleshooting the system, it is sometimes useful to check the flow through the DR impedance. This wizard checks the flow by controlling the PPMS temperature at 20 K, then sealing about 650 Torr of helium mixture in the condenser. The pressure in the condenser then decays as helium flows through the impedance. The rate of change of pressure is a measure of impedance flow.

The flow test is performed at 20 K, rather than room temperature, because the higher density of gas at lower temperature makes the volume flow rate higher. At 300 K, the flow rate is so small that the pressure decay rate in the condenser is too slow, but at 20 K the decay rate is more easily measured. The flow test is not performed at lower temperatures in order to avoid condensation effects.

To check the impedance flow, run the “Check DR Impedance Flow at 20 K” wizard. The software guides you through the following steps:

1. The wizard prompts you to decide whether to perform a flow test or just calculate the flow from previously acquired data. Select the **Flow Test** button in order to perform the flow test.
2. Insert the DR probe into the PPMS sample chamber if the probe is not already inserted.
3. Wait while the wizard purges the chamber and sets the PPMS to 20 K, where the flow test will be performed.
4. Wait while the wizard fills the condenser with gas mixture, then monitors the pressure in the condenser while the flow test is performed. The flow test data will be displayed in PPMS MultiVu.
5. After the test is complete, the wizard prompts you to calculate the flow. You may choose to calculate the flow, or just send the data file to Quantum Design for analysis. Calculate the flow by finding the part of the pressure vs. time curve that is linear and picking two data points, one at each end of the linear region. Enter these two points into the dialog presented by the wizard in order to calculate the flow rate. Record the flow value for future reference and discussion with Quantum Design service personnel.
6. If asked, send the data file, displayed in PPMS MultiVu, to Quantum Design service for further analysis.

After exiting the wizard, you may set a temperature in the usual way. If you started the flow test from room temperature, so that the sample chamber was purged at room temperature, then you may cool the Dilution Refrigerator to low temperatures. However, if you started the test at a temperature less than 295 K, then the purge operation performed by the wizard causes the thermal contact to the condenser to be poor. In this case you should warm up the DR and remove it from the PPMS before attempting to cool it down.

5.2.7 Leak Checking the System

Quantum Design service personnel may ask you to leak check the helium mixture space in your DR in order to troubleshoot certain types of problems. In order to leak check the system, you need a helium leak detector. If you do not have access to a leak detector or if you do not have sufficient expertise with using a leak detector, please contact Quantum Design for assistance.

Before starting this procedure, verify that the system is in the helium-stored configuration with both the “TANK” and “FILL” valves closed. That is, you must have already run the “Secure Gas Mixture” wizard described in section 5.2.5.

If you have access to a helium leak detector, run the “Leak Check System” wizard. The software guides you through the following steps:

1. Hook up a leak detector to the fill access port on the gas handling assembly. You may want to use the provided system pump out hose (Figure 5-1) or an appropriate VCO-type adapter to make the connection to the leak detector.
2. Open the green valve labeled “FILL.”
3. Start the leak detector.
4. Wait while the wizard warms up the turbo pump.
5. The wizard runs the turbo and diaphragm pumps while you leak check the system. Due to the storage of ^4He in the system under normal usage, you may need to wait several hours or overnight in order to achieve low background leak rate needed for good leak checking.
6. Once the leak checking is complete, close the “FILL” valve.
7. Stop the leak detector and disconnect it from the DR.

If you find a leak in the system, the leak must be repaired, and then the leak checking repeated. If you find no leak and are ready to cool down the system, first perform the “Evacuate and Seal” wizard as described in section 5.2.4.

5.2.8 Recharging the Helium Mixture

You may need to periodically recharge the helium mixture in the DR. Quantum Design recommends replacing the mixture when there is less than 600 Torr of mixture when the gas is pumped into the tank. However, you may be able to run the system with less mixture, and doing so will not harm the system.

To recharge the helium mixture, you will need an external tank that is pre-charged with the correct mixture. The external tank is part of the DR Mixture Replacement Kit (part number 4091-175) and is filled with approximately 3.7 liters of 33% He^3 -67% He^4 mixture. This tank connects to the gas handling system through the double sealing connector used when cryocleaning.

You should evacuate all the old mixture out of the system before recharging with the new mixture. To do so, perform the “Evacuate and Seal” wizard with the “TANK” valve open, as described in section 5.2.4.

While it is possible to either add helium to the old mixture or add the full mixture by hand, Quantum Design does not recommend doing so. If you add mixture manually and do not achieve the correct $^3\text{He}/^4\text{He}$ ratio, you may experience problems with the system that are very difficult to troubleshoot.

After you have received a pre-charge tank and have evacuated the old mixture from the system, run the “Recharge the Helium Mixture” wizard. The software guides you through the following steps.

1. The wizard prompts you to make sure the “TANK” valve is open. Do not attempt to perform this operation without opening the “TANK” valve. Doing so could result in a loss of the new mixture when the wizard attempts to pump the mixture from the external tank into the system.
2. Wait while the wizard checks the pressure in the system to make sure that the system has been evacuated.
3. Connect the external tank to the system using the double sealing cryoclean fitting.

4. Wait while the wizard pumps the mixture out of the external tank into the system.
5. Disconnect the tank from the system when prompted to do so.
6. Wait while the wizard measures the quantity of helium mixture in the system after performing the recharging operation.

After completing the wizard, you should perform a cryoclean operation as described in section 5.2.1 before attempting to cool down the system.

5.2.9 Rebuilding the Diaphragm Pump

This procedure must be followed to service the diaphragm pump. If you are replacing the diaphragms, you must have obtained the proper replacement parts from Quantum Design or the pump manufacturer. If there are no leaks into the system and you are only servicing the flapper valves, you should only need to clean the valves with alcohol. In either case, it is strongly recommended that you have a leak detector available for this procedure.

Before starting this procedure, verify that the system is in the helium-stored configuration with both the “TANK” valve and the “FILL” valve closed.

1. Place the Helium-3 insert assembly in a safe place other than the cart. You will need to have full access to the diaphragm pump assembly located inside the cart and the insert will interfere if it is in the cart.
2. Remove the diaphragm pump assembly from the cart and place it on a low work surface or on the floor in front of the cart. To remove it from the cart, you may need to loosen the strain-relief associated with the large black connector running between the insert and pump assembly.
3. Vent the system to atmosphere by opening the “FILL” valve.
4. Unscrew the VCO-type fittings on the two short, flexible hoses that connect the pump to the manifold.
5. Remove either (a) the connecting bar or (b) the bellows hose from the top of the diaphragm pump-head assembly. *Your system has either the connecting bar or the bellows hose.*
 - To remove the connecting bar, unscrew the banjo bolts at either end of the bar. Note the amount of compression in the O-rings (Viton size 2-110) so that you can reinstall them with the right force when the rebuild operation is complete. If you are replacing the diaphragms, you also need to remove the cam cover.
 - To remove the bellows hose, remove the VCO connector fittings at either end of the hose.
6. If possible, lay the pump assembly on one side before removing the first head. There are loose components inside that may fall out if the pump is in its normal upright orientation.
7. Before disassembling each head, note the orientation and location of the two head pieces with respect to the pump body. These parts must go back together in exactly the same configuration.
8. On the head that is facing up, remove the four screws using a 3-mm hex wrench and carefully lift off the silver plate. You should see two rubber disks still stuck to the brown plate. These disks, each with a small hole in the middle, function as flapper valves.
9. Remove the flapper valves and clean them thoroughly in isopropyl alcohol, and then set them aside. If you are replacing the diaphragms, you may discard the flapper valves because new ones are included in the diaphragm repair kit.

10. Carefully clean any debris on the silver plate by using alcohol or another solvent and a cotton swab. Clean the debris out of the holes as well. Set the silver plate aside.
11. Carefully lift the brown plate off and clean it thoroughly with alcohol or another solvent. Be careful not to get any debris into the holes.
12. If you are replacing the diaphragms, lift up the edges of the old diaphragm and unscrew it. Work carefully so that you do not lose the washers on the other side of the diaphragm. Install the new diaphragm using the original washers. If you are not replacing the diaphragms, thoroughly clean the diaphragm surface with alcohol without removing the diaphragm from the pump.
13. Put a small bead of Apiezon H Grease around the outer 2 mm of the diaphragm. Place the brown plate back onto the diaphragm in the original orientation.
14. Place the cleaned, or new, rubber flapper valves back onto the cleaned brown plate in the original location.
15. Put the four socket cap screws in the silver plate and lower it back onto the brown plate, using the screws to precisely align the parts. Verify that the rubber gaskets and O-ring still have a small amount of H Grease for sealing. However, make sure that the flapper valves do not have any grease on them; grease on the flapper valves will cause them to stick. Tighten down the four screws using a “round-robin” technique until the O-ring is compressed and the metal plates are held tightly together.
16. Lay the pump on its other side and repeat the above steps for the second head.
17. Reinstall either (a) the connecting bar or (b) the bellows hose on the top of the diaphragm pump-head assembly. *Your system has either the connecting bar or the bellows hose.*
 - To reinstall the connecting bar, tighten the banjo bolts to compress the O-rings as noted in step 5 (approximately 80 inch-pounds using a torque wrench). Use a small amount of H Grease on the O-rings. If you replaced the diaphragms, reinstall the cam cover first.
 - To reinstall the bellows hose, reconnect the VCO connector fittings at either end of the hose.
18. Reattach the VCO-type fittings that are on the flexible hoses to the manifold.

Installation Instructions

A.1 Introduction

- Section A.2 explains how to install the Dilution Refrigerator option.

A.2 Installing the Dilution Refrigerator Option

Follow these instructions the first time you set up the Dilution Refrigerator on your PPMS. If you have previously set up the DR and need to use it after a period of inactivity, follow the instruction in Section 4.3.

A.2.1 Install the Modular Control System and CAN Network Adapter

You will use the instructions in this section only if a Model 1000 Modular Control System and the driver software have not yet been installed on your PPMS and PC. If your system has already been configured to use Quantum Design CAN modules, you can go to Section 6.2.2.

To install the Model 1000, CAN network adapter, and CAN Manager driver software, refer to the *Model 1000 Modular Control System User's Manual*.

System Verification

After you have completed installation of the Model 1000 and other components, perform a verification of the system:

1. Verify that the power cable has been connected to the Model 1000 and it has been turned on.
2. Verify that the power LED on the front of the Model 1000 is lit green.
3. Verify that the Model 1000 is connected to the CAN adapter on the PC via the CAN network cable.

A.2.2 Change the Model 6000 EPROMs

Note: it is necessary to follow the instructions in this section only if you purchased the DR as an upgrade to your PPMS. If the DR was included in your original PPMS purchase, the correct EPROMs are already installed in your Model 6000.

1. Back up the ROM configuration by running the ROM configuration utility. Do the following:
 - a. Open the **PPMS 32-bit Tools** folder on the PC desktop.
 - b. Run the **Romcfg32** utility.
 - c. Select the Diag (all Configs including above) check box.
 - d. Select the **Read Configuration** button.
 - e. Specify a file name for the configuration you are saving and select **OK**.
2. Turn off the power to the Model 6000 PPMS Controller.
3. Remove the power cord from the Model 6000.
4. Remove the lid from the PPMS electronics cabinet.
5. Remove the lid from the Model 6000.
6. Remove the two EPROMs from the CPU board. When you face the front of the electronics cabinet, the CPU board is the board located furthest to the left.
7. Install the two new EPROMs (revision 1.914 or later) provided with the option into the CPU board. The EPROMs are labeled “PPMS #1” and “PPMS #2.” The CPU board is labeled to indicate the correct placement of the EPROMs. The notch in the EPROMs should line up with the notch in the sockets.
8. Put the cover back on the Model 6000. Place the lid back on the PPMS electronics cabinet.
9. Reinsert the Model 6000 power cord and power up the Model 6000.
10. Run the ROM configuration utility, and send the previously backed-up ROM configuration to the Model 6000.
11. Set the system temperature to 300 K so that the PPMS sample chamber will be ready for the testing you will subsequently perform during the Dilution Refrigerator Installation.

A.2.3 Install the Software

Note: it is necessary to follow the instructions in this section only if you purchased the DR as an upgrade to your PPMS. If the DR was included in your original PPMS purchase, the correct software is already installed on your PC.

Included with the Dilution Refrigerator Option is a software CD. From this CD, install two pieces of software:

1. Install the most recent version of the PPMS MultiVu software (Version 1.4.0 or later) if it is not already installed.
2. Install the DR software by starting the DR software setup wizard and following the instructions.

3. Verify that the DR software is properly installed by starting MultiVu. You should see a new menu item, **Instrument>>Dilution Refrigerator**. When you select this menu item, the **DR Option Control Console** will open.

A.2.4 Insert the Temperature Control Module (Model CM-D)

Note: it is necessary to follow the instructions in this section only if you purchased the DR as an upgrade to your PPMS. If the DR was included in your original PPMS purchase, the module is already installed in the Model 1000.

Follow these instructions to add the Model CM-D DR Temperature Control Module to your Model 1000.

1. Turn off the power to the Model 1000 and disconnect the power cord.
2. Open the lid on the Model 1000.
3. Remove the cover plate for one module bay. You may choose a module bay from either the front or the back row. If a bay is available in the front row, Quantum Design suggests that you choose the front row in order to leave high power, back row, bays open for modules which need the greater cooling provided by the back row.
4. Carefully insert the module into the module bay. Gently tighten the securing screws only until they are finger tight. The faceplate of the module should be flush with the surrounding plates.
5. Reconnect the power cord and turn on the power to the Model 1000.
6. Verify that both the PWR and COP (CANopen Protocol) indicator LEDs on the Temperature Control Module are green (they might be flash red when the power is first turned on, but they should turn green shortly).

A.2.5 Place the Dilution Refrigerator on the Cart

CAUTION!

It is strongly recommended that at least two people handle the gas handling assembly and DR probe while completing the following procedure. The DR probe is very fragile. The thin-walled tubing of the probe shaft can be easily dented or bent, and the turbo pump mounted on the probe is very sensitive to mechanical shocks. The gas handling assembly contains many gas-tight seals that could be compromised if the assembly is dropped or handled roughly.

1. Remove the gas handling assembly and DR probe from the packing crate. You may set the gas handling assembly on the floor. Do not set the DR probe on the floor. Rather, someone should hold the probe until you are ready to place it in the cart.
2. Examine the gas handling assembly. If any electrical connectors were unplugged for shipping, reconnect them.

3. Carefully place the gas handling assembly at the back of the open cabinet area in the DR cart. Position the assembly so that the four rubber feet fit into four of the holes in the floor of the cabinet. The assembly will need to be placed far to the right in the cabinet. Refer to Figure 2-4. Work with care to avoid torquing, crimping, or loosening any of the attached hoses.
4. Attach the tether, which is the black cable running between the gas handling assembly and probe, to the handle of the cart by using the strain-relief clamp located on the back side of the cart handle. To do this, you must pass the probe under the handle so that the tether can run directly up to the clamp from inside the cart. Use the provided 9/64-inch hex wrench to tighten the clamp.
5. Orient the refrigerator probe so that the turbo pump is at the bottom and the probe shaft is at the top. Place the refrigerator probe in the holder in the DR cart. The turbo pump should rest on the rubber mat that is on the floor of the cabinet area. Close the safety latch on the cart.
6. Place the step stool included with the DR option at the base of the PPMS dewar.

A.2.6 Connect the Cables

1. Position the DR cart close to the PPMS and Model 1000 such that the DR probe can easily be inserted into the PPMS.
2. Connect the CAN cable from the rear panel of the Model 1000 to the connector labeled “J1-QD CAN” on the DR gas handling system.
3. Plug the power cable into the DR gas handling system. To reduce the possibility of ground loops, the gas handling system should be plugged into the same power strip as the PC and Model 1000.
4. Make sure the CAN cable from the PC to the Model 1000 is plugged in and the Model 1000 is powered.
5. Connect the temperature cable to the port labeled “JD1-Temperature” on the Model CM-D Temperature Control Module.

A.2.7 Cryoclean the Helium Mixture

The helium mixture often becomes somewhat contaminated during the period between shipment and installation of the Dilution Refrigerator. Therefore, you should cryoclean the gas mixture before attempting to use the system. Follow the procedure “Cryocleaning the Helium Mixture Using the LN₂ Cold Trap” (Section 5.2.1).

A.2.8 Test the Dilution Refrigerator

CAUTION!

Handle the DR probe very carefully. To help prevent the probe from being damaged, stand on the provided step stool when you insert or remove the probe from the PPMS sample chamber. Support the weight of the probe using the turbo pump, not the delicate shaft of the probe. Handle the shaft of the probe only to guide it into the sample chamber.

1. Apply a small amount of Apiezon N Grease to the contact fingers on the condenser tube and the probe shaft. The grease should not be visible but the contact fingers should feel tacky.
2. Inspect the soft silicone O-ring at the turbo pump base. Make sure it is clean and well greased. It may be necessary to clean it and apply a small amount of Apiezon M Grease to ensure a proper seal.
3. Place the step stool next to the PPMS dewar.
4. Set the PPMS to 300 K and wait for it to stabilize there. Wait at least 30 minutes at 300 K before continuing. Vent the PPMS sample chamber.
5. Remove the clamp around the sample chamber access port. Remove the flange from the PPMS access port.
6. Open the safety latch on the Dilution Refrigerator cart and remove the DR probe from the cart. Be sure to hold the DR probe at the turbo pump base. Carefully rotate the DR probe such that the tether is not twisted and the turbo pump is on top.
7. Step onto the step stool and raise the DR probe over the PPMS access port. The black mesh tether and two Fischer connectors on the probe head should be pointing to the right.
8. Slowly insert the DR probe into the PPMS sample chamber keeping the probe well aligned with the chamber. The resistance due to the contact fingers should increase the last 5-6 cm.
9. Make sure the O-ring surface is properly seated onto the PPMS access port flange.
10. Plug in the temperature cable from the Model CM-D Temperature Control Module into the upper Fischer connector, labeled TEMPERATURE, on the right side of the probe head. Plug the sample cable for your measurement option into the lower Fischer connector, labeled SAMPLE.
11. Purge the sample chamber.
12. Run the sequence "DR installation test.seq" to test the temperature performance of the system. If you do not have this sequence, simply set 0.050 K from PPMS MultiVu. In either case, you will need to wait overnight for the system to complete the test.
13. Open the DR log file (press the **View** button on the **Log DR Data** dialog) and verify that the system holds 50 mK. If you ran the installation test sequence, you can also verify that the system controlled at the desired set points by comparing the "Sample Temp." field to the "DR Setpoint" field.

- Section B.2 illustrates the system connections.
- Section B.3 contains pinout table for the wiring connections.

Figure B-1. System cabling/block diagram

B.3 Pinout Tables

Table B-1. Interconnection table for gas handling system CAN cable (Part Number 3091-401)

9-PIN D-Shell Connector at J3 on Model 1000	9-Pin D-Shell Connector at J1 on Gas Handling Module	Description
1	No Connection	-24 VDC
2	2	CAN low
3	No Connection	24 VDC return
4	4	Sync low
5	5	Line sync
6	6	System ground
7	7	CAN high
8	8	Synch hig/Reset
9	No Connection	+24 VDC

Table B-2 Interconnection table for the DR temperature cable (Part Number 3091-150)

25-Pin D-Shell Connector JD-1 at CM-D DR Temperature Control Module	Filter Board in RF Filter Box – Cable Side	Filter Board in RF Filter Box – Connector Side	12-Pin Fischer at Probe Head Labeled Temperature	Description
5	2	1	1	Sample therm. I+
18	1	2	2	Sample therm. I-
6	4	3	3	Sample therm. V+
19	3	4	4	Sample therm. V-
7	6	5	5	Still therm. I+
20	5	6	6	Still therm I-
8	8	7	7	Still therm. V+
21	7	8	8	Still therm. V-
9 and 10	10	9	9	Sample heat I+
22 and 23	9	10	10	Sample heat I-
11 and 12	12	11	11	Still heat I+
24 and 25	11	12	12	Still heat I-
13	13	13	No connection	Analog ground

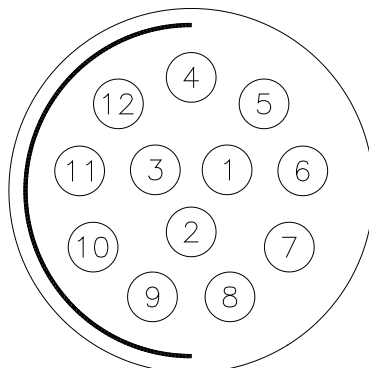
Table B-3 Interconnection table for the DR heat capacity cable (Part Number 3091-620)

9-Pin D-Shell Connector JE-1 at CM-E Heat Capacity Module	Filter Board in RF Filter Box – Cable Side	Filter Board in RF Filter Box – Connector Side	10-Pin Fischer at Probe Head Labeled Sample	Sample Stage Connection	Description
3	2	1	3	3	Platform heater I+
10	1	2	4	4	Platform heater I-
4	4	3	5	5	Platform heater V+
11	3	4	6	6	Platform heater V-
2	6	5	7	7	Platform therm. I+
9	5	6	8	8	Platform therm I-
8	8	7	9	9	Platform therm. V+
15	7	8	10	10	Platform therm. V-
7	13	13	Dewar	No connection	Analog ground

Table B-4 Interconnection table for the DR user experiment cable (Part Number 3091-630)

25-Pin D-Shell Connector at P1-User Bridge or User Electronics	Filter Board in RF Filter Box – Cable Side	Filter Board in RF Filter Box – Connector Side	10-Pin Fischer at Probe Head Labeled Sample	Sample Stage Connection	Description
5	2	1	3	3	Ch1 I+
18	1	2	4	4	Ch1 I-
6	4	3	5	5	Ch1 V+
19	3	4	6	6	Ch1 V-
7	6	5	7	7	Ch2 I+
20	5	6	8	8	Ch2 I-
8	8	7	9	9	Ch2 V+
21	7	8	10	10	Ch2 V-
13	13	13	Dewar	No connection	Analog ground

A. 12-pin Fischer Connector



B. 10-pin Fischer Connector

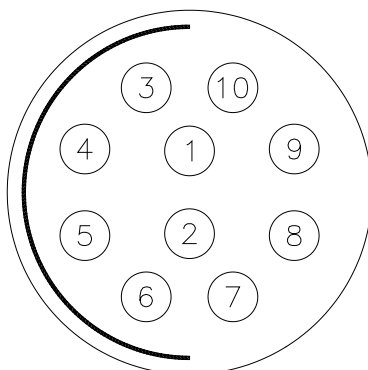


Figure B-2. DR probe connector pin designs

- A) Pin number designations for 12-pin thermometry connector
- B) Pin number designations for 10-pin sample connector

Table B-5 Interconnection table for the DR-ETO cable (Part number 3091-626)

8-Pin Fisher Connector Labeled ETO Head	10-Pin Fisher Connector Labeled DR Probe Head Sample	Sample Stage Connection	Description
1	3	3	Ch1 I+
8	4	4	Ch1 I-
4	5	5	Ch1 V+
5	6	6	Ch1 V-
6	7	7	Ch2 I+
2	8	8	Ch2 I-
7	9	9	Ch2 V+
3	10	10	Ch2 V-

Glossary

Standard Term	QD Part Number	Definition
Cold trap	4092-650-02	Can with molecular sieve used to cryoclean the gas mixture
Concentrated phase		When phase separation occurs, the concentrated phase is the phase that is nearly pure ^3He with almost no ^4He in solution.
Condenser tube		Tube that threads onto the condenser. Five rows of contact fingers contact the PPMS sample chamber. Tube surrounds and protects the delicate dilution unit. Should not be removed without Quantum Design approval.
Counter-flow heat exchanger		Two concentric tubes that connect the still and mixing chamber. Concentrated phase liquid flows down the inner tube, and is cooled by dilute phase liquid flowing up the outer tube.
Dilute phase		When phase separation occurs, the dilute phase is the phase that is mostly ^4He with about 6% ^3He in solution.
DR		Dilution Refrigerator.
Dilution unit		Part of the DR below the condenser, consisting of the still, mixing chamber, and counter-flow heat exchanger. The condenser tube protects this delicate assembly.
Gas handling module	4091-430	Electronic module which controls the gas handling system.
Gas handling system		Assembly consisting of diaphragm pump, manifold, tank, turbo pump controller, and gas handling module.
Helium mixture		The mixture of ^3He and ^4He gas which is used to cool the DR.
Impedance		The flow impedance between the condenser and still coil on the dilution unit.
Mixing chamber		The lower pot in the DR where phase separation and dilution cooling occur.
Model 1000		Control center tower for CAN modules. Model CM-D Temperature Control Module resides here.
Model CM-D or Temperature Control Module		The DR Temperature Control Module which measures thermometers and applies heat for feedback control
Probe		The section of the system inserted into the PPMS sample chamber.
Radiation shield		Tube which screws onto the condenser tube and protects the sample stage. This tube is the bottom end of the DR when it is in the PPMS.
Sample mount	4091-610 4085-264	The hardware where the user mounts the sample to be measured. The pins on the sample mount plug into the sockets on the sample stage, where the 8 experimental wires are accessed.
Sample mounting pins	4091-615	Pins which are used to immobilize the sample stage when mounting or unmounting sample mounts. This prevents damage to the dilution unit.

Glossary

Sample stage	4091-300	Part of the DR where the sample mounts plug in. Contains the sample thermometer, thermal standoff, and 8-pin interface for the sample wiring.
Still		The upper pot in the DR where ^3He gas is evaporated from the dilute phase.

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