

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



Physical Property Measurement System

Helium-3 Refrigerator System User's Manual

Part Number 1092-100, D7

Quantum Design

10307 Pacific Center Court
San Diego, CA 92121
USA

Technical support (858) 481-4400
(800) 289-6996
Fax (858) 481-7410

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U.S. Patents

5,311,125 Magnetic Property Characterization System Employing a Single Sensing Coil Arrangement to Measure AC Susceptibility and DC Moment of a Sample (patent licensed from Lakeshore)
5,647,228 Apparatus and Method for Regulating Temperature in Cryogenic Test Chamber
5,798,641 Torque Magnetometer Utilizing Integrated Piezoresistive Levers

Foreign Patents

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

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P R E F A C E

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) Helium-3 Refrigerator System option. This manual explains how to install the Helium-3 system, discusses the hardware and software that are unique to the Helium-3 system, and explains basic operation and maintenance procedures for the Helium-3 system.

P.3 Contents of the Manual

- Chapter 1 introduces the Helium-3 system and discusses the theory of operation for the Helium-3 system.
- Chapter 2 discusses the hardware used with the Helium-3 system.
- Chapter 3 discusses the Helium-3 system software.
- Chapter 4 describes the basic operation of the Helium-3 system.
- Chapter 5 describes the basic operation of Helium-3 in Dynacool.
- Chapter 6 contains a troubleshooting guide and explains how to perform common maintenance procedures.
- Appendix A explains how to install the Helium-3 system.
- Appendix B illustrates Helium-3 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 or on the Model 6000 PPMS Controller LCD screen.
- File >> Open** The >> symbol indicates that you select multiple, nested software options.
- STATUS** Bold text and all capital letters distinguish the names of keys located on the front panel of the Model 6000 PPMS Controller.
- .dat** The Courier font distinguishes characters you enter from the PC keyboard or from the Model 6000 PPMS Controller front panel. The Courier font also distinguishes code and the names of files and directories.
- <Enter>** Angle brackets distinguish the names of keys located on the PC keyboard.
- <Alt+Enter>** A plus sign connecting the names of two or more keys distinguishes keys you press simultaneously.
- Note** Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances.

CAUTION!

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



WARNING!

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



WARNING!

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

Theory of Operation

1.1 Introduction

This chapter contains the following information:

- Section 1.2 presents an overview of the Helium-3 system.
- Section 1.3 discusses the theory of operation for the Helium-3 system.
- Section 1.4 discusses the Helium-3 temperature control.

1.2 Overview of the Helium-3 System

The Quantum Design Helium-3 Refrigerator System option (Model P825) extends the minimum temperature of the Physical Property Measurement System (PPMS) or Physical Property Measurement System DynaCool (DynaCool) below 0.4 K. The Helium-3 system is integrated seamlessly into the base PPMS or DynaCool platform, and its operation is designed to be completely transparent to the PPMS or DynaCool measurement application software.

The Helium-3 Refrigerator system is entirely self-contained. The Helium-3 insert, pumping lines, electrical cables, and diaphragm or scroll pump are incorporated into a small cart that can be rolled to any location when the system is not in use. Newer Helium-3 systems use a scroll pump to achieve better pumping efficiency and lower leak rate compared to the backing pump used on older systems. The lower leak rate minimizes ^3He gas loss. The efficient design of the gas-handling system requires less than 1 liter of replacement gas per year under heavy usage. This is made possible by using a gas-handling system that is sealed and uses oil-free pumps for gas circulation.

The Helium-3 system on PPMS platform is compatible with the PPMS Resistivity, Electrical transport, and Heat Capacity options. The Helium-3 system on DynaCool platform is compatible with the Electrical transport and Heat Capacity options.

Once the samples are mounted on the Helium-3 insert and installed in the PPMS or DynaCool sample chamber, the temperature control is handled automatically down to below 0.4 K without the need to learn new measurement control software.

Note: First time installation instructions for the Helium-3 system, please refer to Appendix A.

1. For PPMS platform, please refer to Appendix A.
2. For DynaCool platform, please refer to Appendix B.

1.2.1 System Requirements

The following components are required to operate the Quantum Design Helium-3 Refrigerator System:

- PPMS Resistivity option with enhanced user bridge board
- PPMS Continuous Low-Temperature Control (CLTC) option
- PPMS High-Vacuum option

The High-Vacuum option is an integral component of the PPMS Heat Capacity option.

1.2.2 System Hardware

The Helium-3 system hardware consists primarily of the insert assembly, the gas-handling assembly, and a cart that holds both assemblies.

1.2.2.1 HELIUM-3 GPIB VERSION

A special controller card in the Model 6000 PPMS Controller provides the necessary control signals. Also, sample-mounting hardware for the Resistivity, Electrical Transport, and Heat Capacity options allows these options to be used with the Helium-3 system.

1.2.2.2 HELIUM-3 CAN VERSION

Helium-3 control module CM-P (4101-455) in the CAN tower provides all the necessary power and logic to drive the Helium-3 option. Also, sample-mounting hardware for the Resistivity, Electrical Transport, and Heat Capacity options allows these options to be used with the Helium-3 system.

Chapter 2 discusses the Helium-3 hardware in detail.

1.2.3 System Software

The software provided with the Helium-3 system consists of a main temperature control module, diagnostics utilities, and maintenance utilities. The Helium-3 Control Console application must be running on the PC in order to enable temperature control with the Helium-3 insert.

Chapter 3 discusses the Helium-3 software architecture in detail.

1.3 Theory of Operation

A helium-3 refrigerator cools to below 0.4 K by pumping on a pot of liquid ^3He . This isotope of helium condenses at slightly above 3 K at 1 atmosphere. Evaporative cooling to below 0.4 K can be achieved if the pressure above the liquid is maintained in the millitorr range and there is sufficient thermal isolation from the warmer surroundings.

With the PPMS Helium-3 system, the cooling power for condensing ^3He is provided by the cold walls of the PPMS sample chamber. A variable-speed turbo pump mounted at the top of the Helium-3 insert and small diaphragm or scroll pump provides the low pressure needed for cooling. Thermal isolation is provided by sealing the insert of the probe into the PPMS or DynaCool sample chamber, which is maintained at high vacuum by using the PPMS High-Vacuum system.

To provide continuous cooling below 0.5 K, the pot is continuously refilled using a very small capillary, or impedance that feeds liquid ^3He at a few hundred torr from the 2-K condenser to the colder pot that is pumped to a few millitorr.

The 2-K liquid flowing into the pot from the impedance constitutes a small heat leak into the pot. Hence the system is not able to achieve the lowest possible temperature while filling the pot. To achieve the base temperature of the instrument, the gas-handling system has been designed to shut off the flow of recondensing gas to the impedance once sufficient liquid has collected in the pot. In this “one-shot” mode, a base temperature of well below 0.4 K can be achieved for over an hour before the pot must be refilled. A ^3He reservoir tank provides the necessary volume of gas in the system to completely fill the pot with liquid when the one-shot mode is needed.

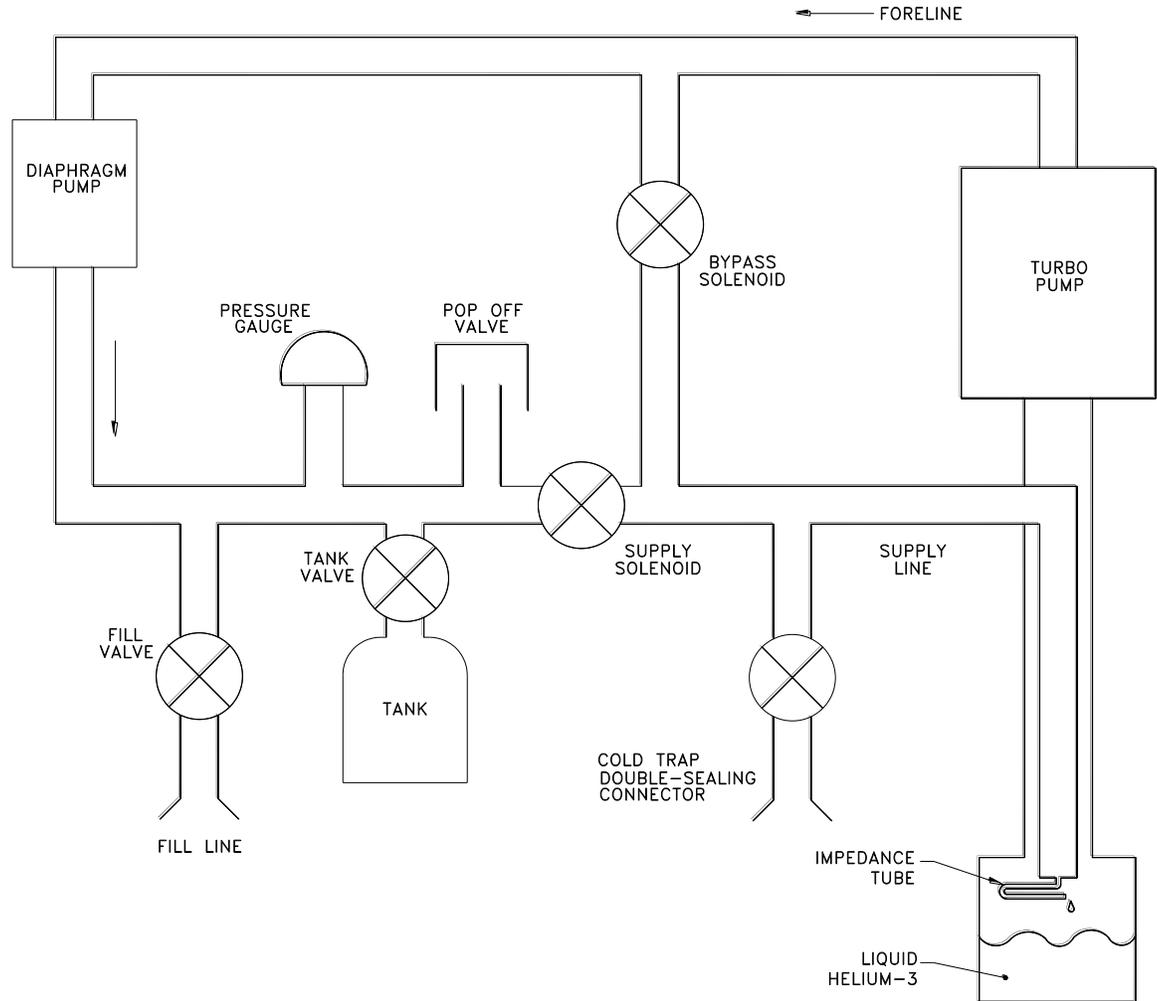


Figure 1-1. Helium-3 System Schematic

User samples are mounted on special sample mounts that plug into a sample holder connected to the pot. The thermal contact between the sample holder and pot is somewhat weak so that the temperature of the holder can be raised using a heater on the holder and an integrated thermometer for control. The standard PPMS temperature control algorithms provide temperature control at higher temperatures.

1.4 Temperature Control

The Helium-3 insert has been designed specifically for cooling samples below 2 K by pumping on a bath of liquid ^3He . However, since it can be inconvenient to remount samples for measurement at

higher temperatures, the Helium-3 system has been designed to provide temperature control from the lowest temperature continuously up to 350 K. To cover such a wide temperature range, several operating modes are used. Table 1-1 lists each of the operating modes and corresponding temperature ranges. The transitions between the operating modes are completely automated through the use of the control software. Automatic initiation of the PPMS High-Vacuum system is also performed when it is needed for proper operation at pumped ^3He temperatures.

Table 1-1. Operating Modes

SAMPLE TEMP	PPMS TEMP	COOLING SOURCE	HEATING SOURCE	CHAMBER STATE	DESCRIPTION
20.00–350.00 K	20.00–350.00 K	PPMS Sample Chamber	PPMS Sample Chamber	Vented or High Vacuum	High Temp
4.00–20.00 K	4.00–20.00 K	PPMS Sample Chamber	PPMS Sample Chamber	High Vacuum	Low Temp Sys Heater
2.50–4.00 K	1.80 K	PPMS Sample Chamber	Helium-3 Stage Heater	High Vacuum	No Cooling, Helium-3 Heater
0.45–2.50 K	1.80 K	Pumped ^3He	Helium-3 Stage Heater	High Vacuum	Circulating Mode
0.35–0.45 K	1.80 K	Pumped ^3He	Helium-3 Stage Heater	High Vacuum	One-Shot Mode

1.4.1 High-Temperature Standard Mode (20–350 K)

In this temperature range, temperature control is very similar to the standard PPMS sample chamber temperature control. Both cooling and heating are provided through the PPMS sample chamber with feedback from the thermometer located on the Helium-3 sample platform. The sample chamber may be in any state (vented, purged, high vacuum, and so on).

When the sample chamber is at high vacuum, thermal contact between the sample chamber walls and the Helium-3 insert is relatively poor. This can result in a much slower thermal response while controlling temperature. The system software automatically adjusts the time-constants in the temperature control routines when the chamber is switched between the high-vacuum state and another state. To achieve the most rapid cool-down, it is recommended that cool-down be initiated with the sample chamber vented or purged. The only exception to this would be during heat capacity measurements where high vacuum is needed for the measurement itself.

Between 20 and 285 K, the system is susceptible to developing plugs from gas contaminants in the Helium-3 space. To address this problem, the diaphragm and turbo pumps are run with the bypass solenoid open and supply solenoid closed to remove most of the gas from the probe. Once the probe is below 20 K, the threat of plugging is reduced by the cold-trapping of the contaminants.

1.4.2 Low-Temperature Standard Mode (4–20 K)

Below 20 K, the sample chamber must be maintained at high vacuum even when high vacuum is not needed for the specific measurement. It is necessary to initiate high vacuum at 20 K to avoid condensing helium or saturating the charcoal at the bottom of the probe. If the sample chamber is inadvertently vented while below 20 K, the control software issues a warning and then attempts to reactivate the High-Vacuum system by sending the temperature back up to 20 K.

Like high-temperature standard mode, low-temperature standard mode uses the PPMS sample chamber for both heating and cooling. The only real difference between the two modes is the requirement that the chamber be maintained at high vacuum.

1.4.3 No-Cooling Mode (2.5–4 K)

From 2.5 to 4 K, it is important to avoid condensing ^3He in the pot. This is because the heat that is released and absorbed by the ^3He condensing and evaporating makes temperature control very sluggish. To address this issue, the diaphragm and turbo pumps are run with the bypass solenoid open and the supply solenoid closed so that all ^3He is pumped away from the cold end of the probe. To control temperature, the PPMS sample chamber is maintained below 2 K and the heater located on the Helium-3 sample stage is used to raise the temperature up to the set point.

When entering this state from lower temperatures, the pot is first evacuated to ensure that all condensed ^3He is removed.

1.4.4 Circulating Mode (0.5–2.5 K)

Once below about 2.5 K, cooling is provided by circulating ^3He . In this mode, the supply solenoid is open and the bypass solenoid is closed. Evaporative cooling takes place in the pot by pumping with the turbo pump. The pot is continuously refilled through the impedance. The turbo pump speed is adjusted depending on the temperature set point and the heat load.

At temperatures below about 1 K, liquid ^3He collects in the pot. The control software determines the level of liquid by monitoring the gas pressure at the tank. Once the pressure is low enough, indicating a full pot, and if the temperature set point is too low to be achieved in the circulating state, the system starts the one-shot mode as described in Section 1.4.5. Generally the pressure must be below about 150 torr to enter one-shot mode.

1.4.5 One-Shot Mode (0.35–0.5 K)

While in the circulating state, the base temperature is limited to about 0.45 K by the heat load created by the 2-K liquid flowing into the pot from the impedance. To achieve the ultimate base temperature, the system can be operated in a one-shot mode, where the supply of ^3He to the impedance is cut off. Without the warm liquid flowing into the pot, the system can attain temperatures below 0.4 K. In this mode, the supply solenoid is closed and the bypass solenoid is open.

Operation in one-shot mode is limited to about 90 minutes before the pot must be refilled, as the pot is not refilled continuously. To determine when the pot must be refilled, the control software monitors the pressure in the tank. When the pressure increases to a specific value, the system automatically switches back into circulating mode to allow liquid ^3He to collect in the pot. The precise value of this crossover pressure is set when the system is charged with ^3He gas. It is remeasured whenever the cyrocleaning procedure is performed (see Section 6.3.1). The system automatically switches back into one-shot mode once the pressure falls to the appropriate value, indicating adequate liquid in the pot.

1.4.6 Standby Mode

When you first start the control software, the Helium-3 temperature control engine automatically enters standby mode. In this state, both valves are open and the circulating pumps are off. The system remains in standby mode until a new temperature set point is specified from the PPMS MultiVu application or from the Model 6000 PPMS Controller. At this point, the control software initiates a system test and then selects one of the operating modes described above.

1.4.7 Probe-out Mode

Whenever the Helium-3 insert is removed while the control software is still running, the system enters the probe-out mode. In this mode, the standard PPMS temperature control engine takes over and the temperature of the sample chamber is controlled as if the Helium-3 option were not present. When the system is in probe-out mode, the control software automatically takes over once the Helium-3 insert is installed in the sample chamber again. A micro-switch located near the head of the insert is used to indicate the probe-out condition.

Hardware

2.1 Introduction

This chapter contains the following information:

- Section 2.2 discusses the Helium-3 insert assembly.
- Section 2.3 discusses the backing pump and gas-handling assembly.
- Section 2.4 discusses the Model CM-P Helium-3 Control Module.
- Section 2.5 discusses Model 1000 (Modular Control System).
- Section 2.6 discusses CAN Computer Interface Kit.
- Section 2.7 discusses the liquid nitrogen cold trap assembly.
- Section 2.8 discusses the Model 6000 PPMS Controller.
- Section 2.9 discusses option-specific Helium-3 hardware.
- Section 2.10 discusses other accessories included with the Helium-3 system.

2.2 Helium-3 Insert Assembly

The Helium-3 insert assembly (Figure 2-1) functions as the sample holder. Samples are mounted on measurement-specific sample mounts that are plugged into the end of the insert assembly. The whole assembly is then plugged into the PPMS sample chamber.

2.2.1 Thermal Contact

To provide the necessary thermal isolation between the ^3He pot and the surrounding environment, the PPMS sample chamber is maintained at low pressure by using the PPMS High-Vacuum option. The vacuum seal is provided at the top flange of the sample chamber by an O-ring under the turbo pump assembly. The system automatically handles the evacuation of the sample chamber when necessary. When the system is operated below about 4 K, evacuation of the sample space is facilitated by cryopump-grade charcoal attached inside the sample housing at the bottom of the insert assembly.

The refrigerator probe shaft of the insert assembly is made of a very thin, stainless steel tube to reduce the static heat load on the PPMS due to the thermal conductance of the shaft. Contact

fingers on the outside of the insert assembly ensure that the temperature profile of the insert matches that of the PPMS sample chamber. This provides precooling of the returning ^3He gas, as well as minimizing the differential thermal contraction of the insert relative to the sample chamber.

To provide the necessary cooling power for condensing ^3He gas, additional contact fingers are located around the sample mount housing.

Thermal isolation of the pot is necessary for proper operation at liquid ^3He temperatures. However, at higher temperatures, better thermal contact is needed for faster temperature control. To satisfy both requirements, a special graphite-loaded polymer is sandwiched between the cooling pot and the insert housing. Since the sample is in thermal contact with the pot, polymer provides an additional thermal path to the sample chamber walls. The material has the property of being thermally resistive at low temperatures and relatively thermally conducting at higher temperatures.

2.2.2 Turbo Pump

To provide a sufficiently low pressure for evaporative cooling, a turbo pump is mounted directly on top of the insert assembly. A relatively small pumping line for backing the turbo pump runs from the insert assembly to the backing pump/gas-handling system. Variable speed operation of the turbo pump accommodates variations in the ^3He gas flow rate. These variations result from different evaporation rates under different thermal loads on the pot.

When heat capacity measurements are running at low temperatures, mechanical vibrations can produce unwanted heating of the Heat Capacity sample mount. To reduce this effect with the Helium-3 system, the natural mechanical resonances of the turbo pump are mapped out when the instrument is calibrated at the factory. The turbo pump is then operated at only those speeds that exhibit minimal vibrations. Furthermore, a vibration attenuation mechanism is integrated into the top of the insert assembly. This mechanism consists of a stainless steel bellows connecting the turbo pump to the probe shaft. A black rubber support surrounds the bellows and provides damping and structural support.

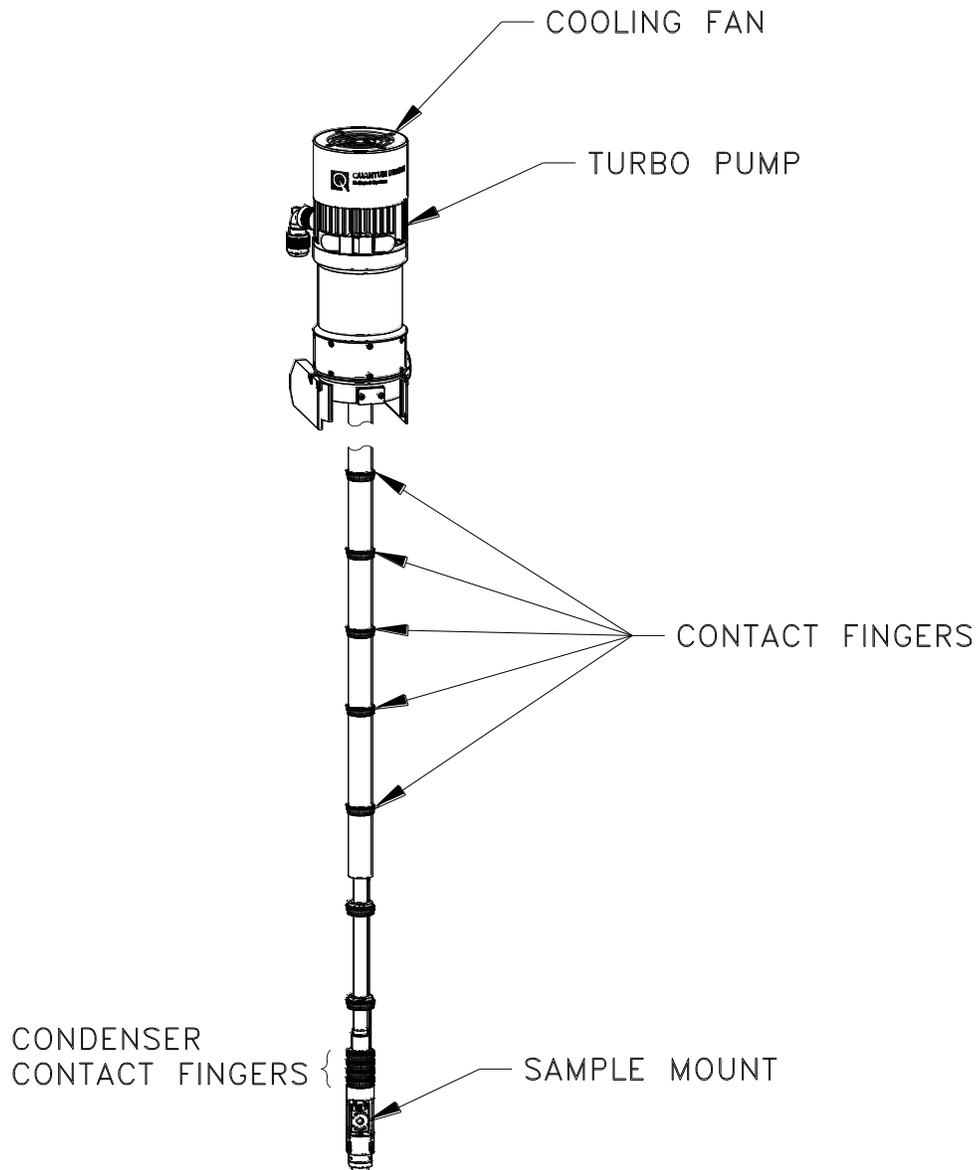


Figure 2-1: Helium-3 Insert Assembly

2.2.3 Helium-3 Pot and Sample Holder

The ^3He gas enters the insert through the connector on the back side of the probe head. A helical tube runs down the inside of the main shaft of the insert and pre-cools the gas as it enters the condenser. Condensed liquid then flows into the pot at the lowest point in the circulation path. Cooling occurs when the liquid in the pot is pumped. The ^3He exits the pot by evaporation and returns to the turbo pump, where it leaves the insert assembly through the back of the turbo pump.

A short thermal standoff attaches the sample holder to the pot. The thermal link is designed to provide adequate cooling power with the added capability to perform rapid temperature control by raising the temperature of the holder above the temperature of the pot through the use of a small heater. A calibrated thermometer is located in the sample holder. User samples are

mounted to the sample holder through the use of measurement-specific sample mounts that plug into the eight-socket connector on the sample holder assembly.

Two sample holders are available to provide different orientations. The vertical sample holder is oriented so that the magnetic field of a longitudinal magnet is in the plane of the holder, while the horizontal sample holder is oriented so that the magnetic field of a longitudinal magnet is perpendicular to the sample holder. The two holders accept the same option-specific sample mounts (see Section 2.9). For systems with a transverse magnet, the vertical sample holder is designed to be loosened, rotated, and retightened to provide different orientations with respect to the field (see Section 4.4).

- Helium-3 insert with a vertical sample holder is on the left.
- Helium-3 insert with a horizontal sample holder is on the right.

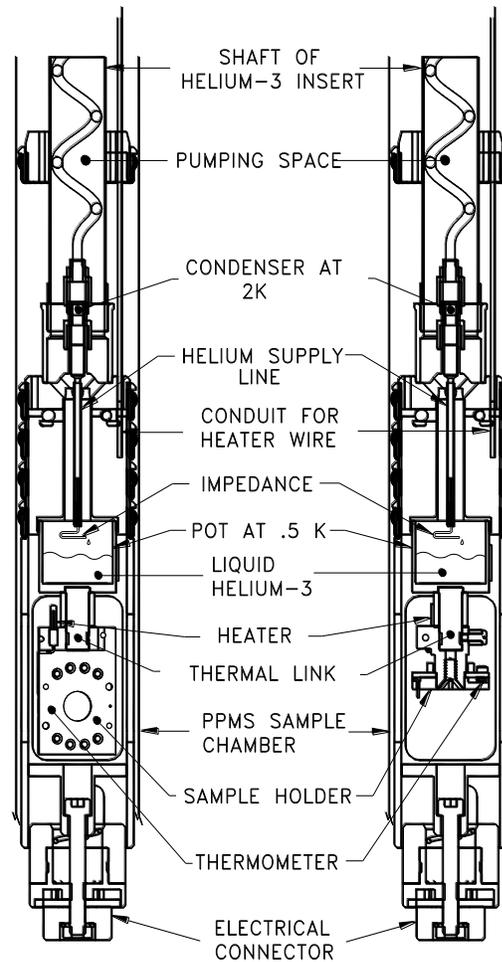


Figure 2-2: Cut-away view of two Helium-3 insert assemblies installed in PPMS sample chambers.

2.2.4 Electrical Connections to Sample

The sample-holder thermometer and the eight sample measurement wires are contacted through the spring-loaded connect or located at the bottom-most point on the insert. This connector, which is designed to engage the 12-pin connector at the bottom of the PPMS sample chamber, is spring-loaded to accommodate slight variations in the vertical position of the probe. The proper orientation for the connector is set by the alignment brackets located on the sides of the turbo pump.

The standard PPMS sample chamber wiring is used to bring the signals out of the dewar via the gray Lemo-type connector. The heater used to raise the temperature of the sample holder is wired-up through a separate connector located on the back side of the insert head.

In the GPIB version of He3, the thermometer channel is read by channel 4 of the user bridge board using Helium-3 experiment cable (3092-359-01/02). The resistance measurements are performed with the standard user bridge cable (part number 3084-003). When performing heat capacity measurements, the standard heat capacity cable (part number 3085-100) is used, and it places the thermometer signal on channel 4. When performing Electrical Transport measurements, a special ACT/Helium-3 cable (part number 3084-518) is used, and it also places the thermometer signal on channel 4. For more information about the signal wiring any of these options use with the Helium-3 system, please refer to the appropriate option manual.

In the CAN Version of He3, the thermometer channel is read by a dedicated resistance bridge in Helium-3 module (4101-553) using Helium-3 experiment cable (3092-359-01/02) through a four pin Lemo-type connector on the module's front panel. The resistance measurement is performed with the bridge board with the standard user bridge cable (part number 3084-003). When performing heat capacity measurements, the standard heat capacity controller cable (part number 3085-110) is used. When performing Electrical Transport measurements, a special ACT/Helium-3 cable (part number 3084-518) is used. For more information about the signal wiring any of these options use with the Helium-3 system, please refer to the appropriate option manual.

2.3 Gas-Handling Assembly

The Helium-3 insert assembly is connected to the gas-handling system by two small gas lines and various electrical cables. The gas-handling system is normally located in the cart that is designed to hold the Helium-3 insert when the insert is not in use. The basic components of the gas-handling system are the backing or scroll pump, tank, gas manifold, and turbo pump controller.

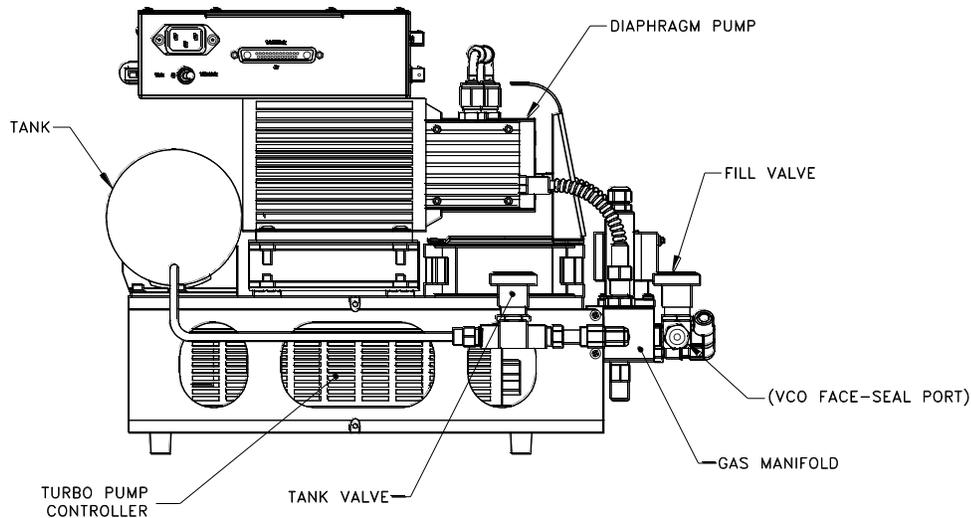


Figure 2-3: Backing Pump and Tank Assembly

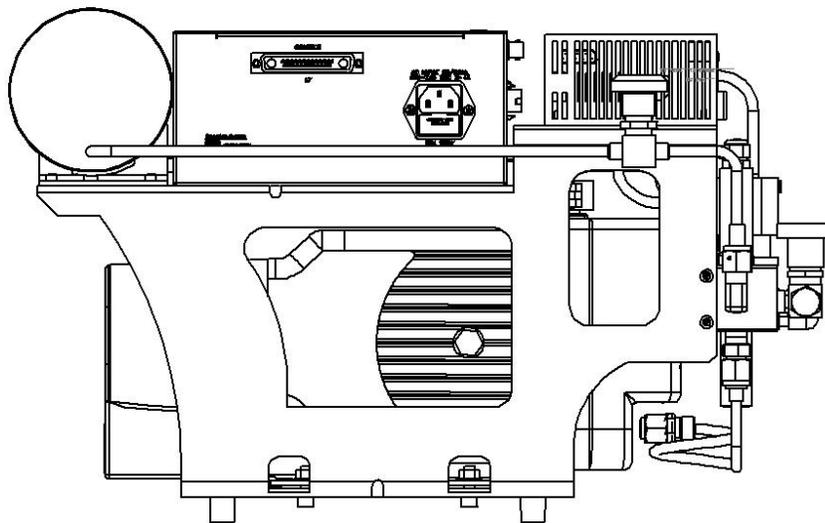


Figure 2-4: Scroll Pump and Tank Assembly

2.3.1 Backing or Scroll Pump and Tank Assembly

The backing or scroll pump maintains the outlet pressure from the turbo pump at a few torr. This is necessary for proper operation of the turbo pump. Both the backing and scroll pump uses no lubricants and employs hermetic seal design in which the motor and bearing are outside the vacuum space allowing full isolation of circulating gas which avoids contamination of ^3He gas in the system. The older version of the Helium-3 system uses a backing pump in the gas handling system. In the new version of Helium-3 system, the backing pump is replaced with a scroll pump in the gas-handling system, to have a better pumping speed (35 l/m), base pressure 750 miltorr and leak rate $\leq 1 \times 10^{-6}$ std-cc/sec helium. During normal operation, the outlet of the backing or scroll pump exhausts the ^3He gas back to the insert assembly.

The tank is also attached to the outlet of the backing or scroll pump to provide a volume for the gas to expand into as the system condenses various amounts of liquid into the pot. The tank also serves as storage for the ^3He gas during maintenance operations. For this purpose, a manual valve is located on the tank to seal off the tank from the rest of the system.

2.3.2 Gas Manifold

While the system operates in a standard circulating mode, the gas flows in a closed loop through the impedance at the bottom of the probe and through the pumps, past the tank, and back into the insert. Another mode of operation shuts off the flow of helium to the impedance to achieve lower temperatures in a one-shot mode. To switch back and forth between these two modes of operation, two solenoid valves, located on the manifold block, are used.

A gauge, which measures the pressure of the gas in the tank, is also located on the manifold block. This gauge allows the temperature control software to infer the level of liquid in the pot as well as diagnose certain fault conditions.

An access port and manual “FILL” valve are also located on the manifold block. The VCO-type, face-seal port is used for adding additional ^3He gas to the system. A manual “CRYO CLEAN” valve is also located on the manifold to secure the ^3He gas leaking through the quick seal connector. A special cryocleaning hose is provided to allow the system to be pumped out by using the PPMS sample chamber as a pumping station.

2.3.3 Electrical Distribution Box and Turbo-Pump Controller

The electrical distribution box and turbo-pump controller are located on the top level of the assembly. A step-up/down toroidal transformer in electrical distribution box provides power to the scroll pump. Special firmware is designed specifically to operate the turbo pump at different speeds. The controller is connected to the turbo pump via a multi-conductor cable located inside the probe tether. Operation of the turbo pump is fully automated.

In Helium-3 GPIB version, communication with the controller is achieved through a serial port that connects to the back of the computer.

In Helium-3 CAN version, communication with the controller is handled by the Helium-3 CAN module electronics through a serial cable.

2.3.4 Cart and Step Stool

The Helium-3 system is incorporated into a cart. When in operation, the backing pump assembly is located inside the cart. A holder mechanism integrated into the cart is used for storing the Helium-3 insert when the insert is not in use. The holder is also convenient for mounting samples onto the end of the insert. The cart can be easily rolled out of the way when it is not used.

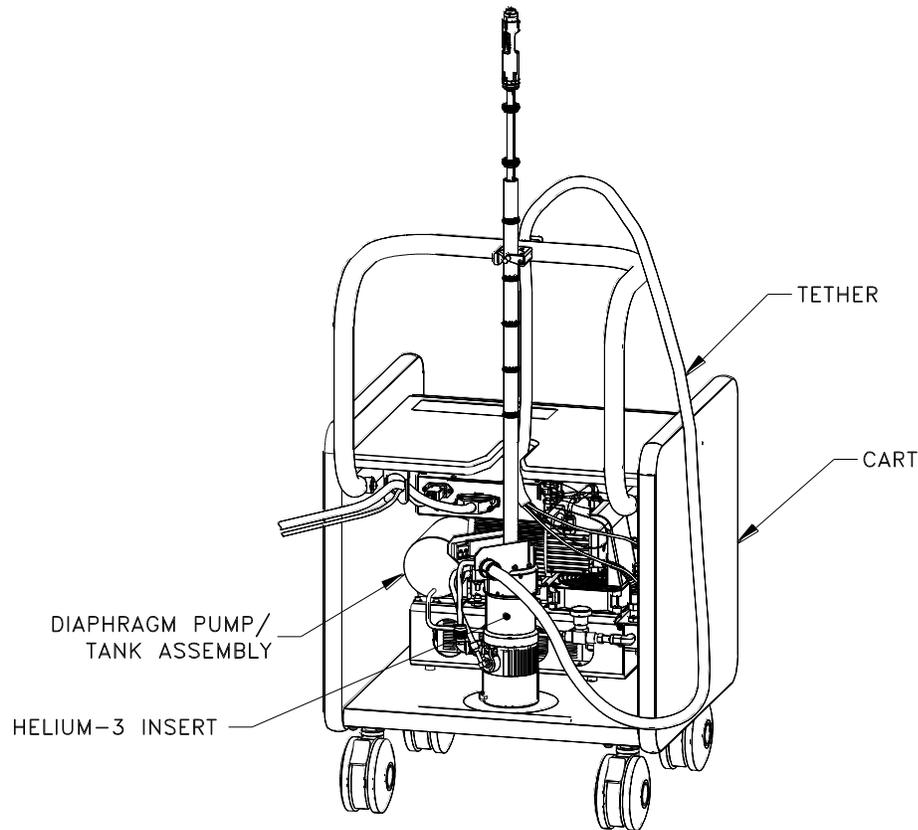


Figure 2-5: Helium-3 Cart Assembly

A step stool is also provided with the Helium-3 system. It is strongly recommended that the stool be used during insertion and removal of the Helium-3 insert from the PPMS sample chamber. The shaft of the probe on the insert is extremely fragile, and the turbo pump is quite heavy by comparison. This combination makes the insert particularly susceptible to damage during handling. Although there is nothing special about the stool, it is included with the system to emphasize the need to handle the insert with care.

2.4 Model CM-P Helium Control Module

The Model CM-P Helium-3 CAN Module (Figure 2-7) provides all of the necessary electronics to read and control temperature on the Helium-3 system. The module uses feedback control to maintain temperature set points received from the user or the control software. The module has

one dedicated resistance bridge for the Helium-3 thermometer and one current source for the Helium-3 heater. (Helium-3 heater and thermometer are on the Helium-3 sample stage vertical/horizontal). The module also energizes the solenoid valve and pressure gauge in the Gas Manifold (refer to section 2.3.2) as required by the Helium-3 software (or) user through the Helium-3 GasMon.

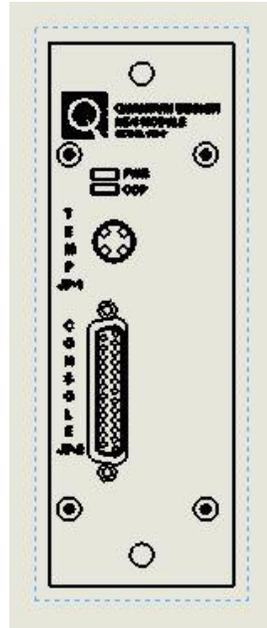


Figure 2-6: Helium-3 Control CAN Module

2.5 Model 1000 (Modular Control System)

The model 1000 modular control system is a general-purpose chassis (Figure 2-8) that house, 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-P helium-3 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.

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

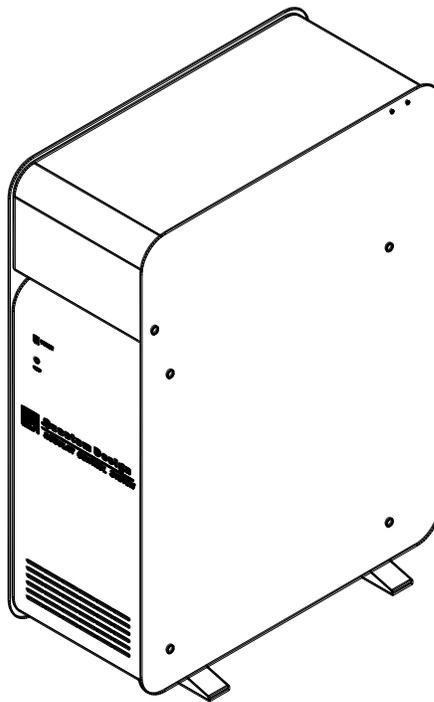


Figure 2-7: Model 1000 Modular Control System

2.6 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).

2.7 Liquid Nitrogen Cold-Trap Assembly

The Liquid Nitrogen Cold Trap removes contaminants from the ^3He gas. As described in Section 6.3.1, “Cryocleaning the Helium-3 Gas Using LN2 Cold Trap,” a wizard leads you through the cryocleaning process. Note that when the system is heavily used, you should perform the cryocleaning procedure every two weeks or so.

2.7.1 Cold Trap

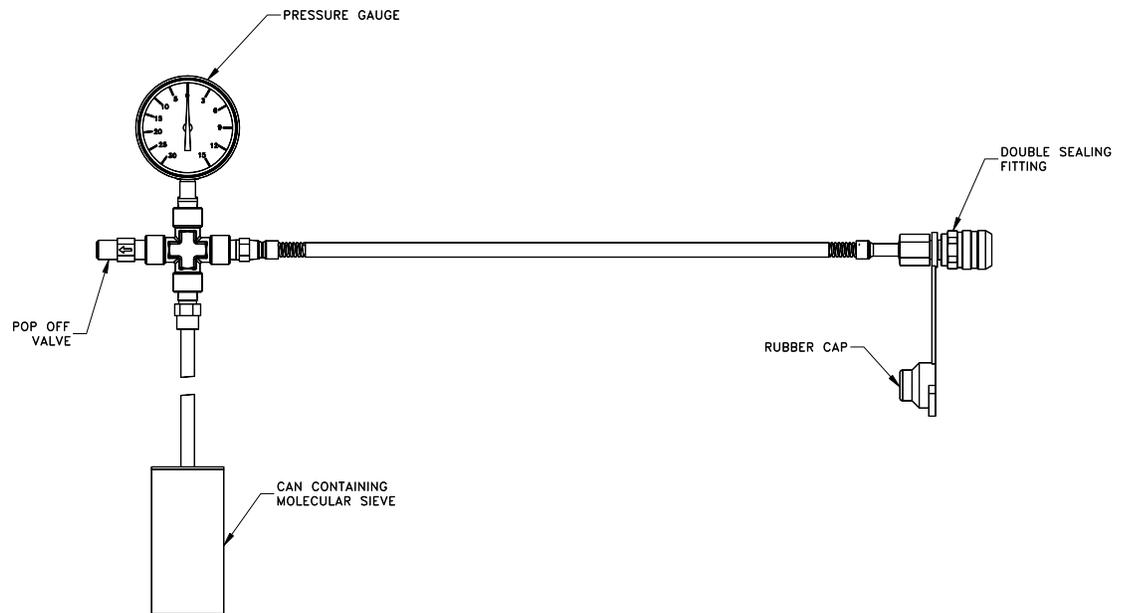


Figure 2-8: Cold Trap

The cold trap contains molecular sieve material in the CAN portion at the bottom of the assembly. When cooled to liquid nitrogen temperature, the molecular sieve readily absorbs any gas or vapor other than helium. This removes contaminants from the ^3He .

A pressure gauge determines residual gas in the assembly. If the gauge reads to the right of zero, the pressure inside the cold trap is greater than ambient pressure. In this case, remove the gas from the cold trap using the procedure “Regenerating the Cold Trap” (Section 6.3.2). 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 this is not the case, the cold trap probably has a leak.

The double sealing fitting on the cold trap connects and disconnects the trap to the Helium-3 gas manifold without letting in atmospheric gases as the fitting is completely sealed when not connected to its mate. When the fitting is not use, place the rubber cap on it to protect it from dirt. Each time you connect the cold trap to the Helium-3 gas manifold, apply a thin film of Apiezon M-grease to the surface of the fitting and its mate on the gas manifold. The grease will lubricate the o-rings in the connectors and prevent leaks.

To prevent a dangerous overpressure condition, a pop-off valve is attached to the cold trap.

2.7.2 Trap Regeneration Fitting

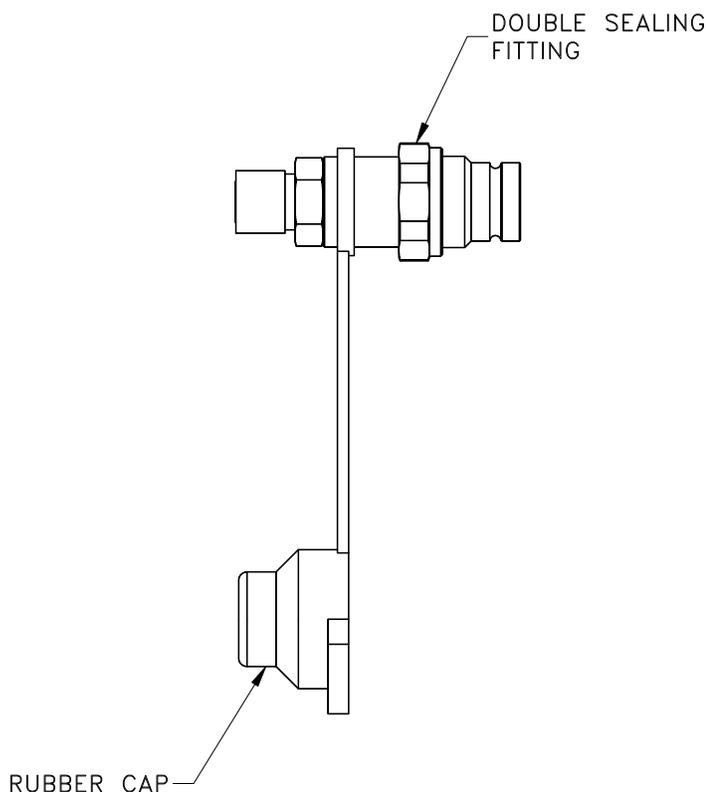


Figure 2-9: Trap Regeneration Fitting

The 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 cryocleaning hose, which is shown in Figure 5-1. The cryocleaning 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 6.3.2). Place the rubber cap on the double sealing fitting when not in use to protect the fitting from dirt.

2.7.3 Liquid Nitrogen Dewar Flask

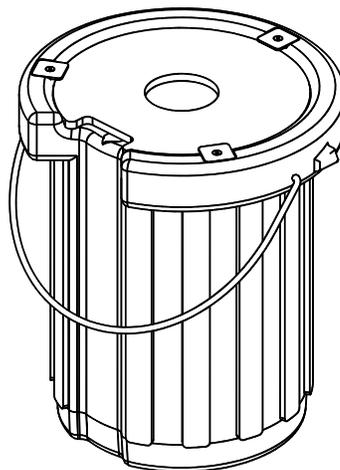


Figure 2-10: Liquid Nitrogen Dewar Flask

The dewar flask is a well-insulated container for liquid nitrogen. The hole in the top of the dewar flask allow the cold trap into the liquid nitrogen.

2.8 Model 6000 PPMS Controller

The expansion circuit board (part number 3076-015) located in the Model 6000 PPMS Controller provides the necessary control signals for the system. This special circuit board drives the two solenoid valves, controls the pressure gauge, and also has a precision current source for driving the sample heater on the sample holder.

Using a resistive temperature sensor below 1 K requires readout electronics that use very little measuring power. The enhanced user bridge board (part number 3076-050) uses a precision balanced current drive to achieve the required sensitivity with minimal self-heating in the temperature

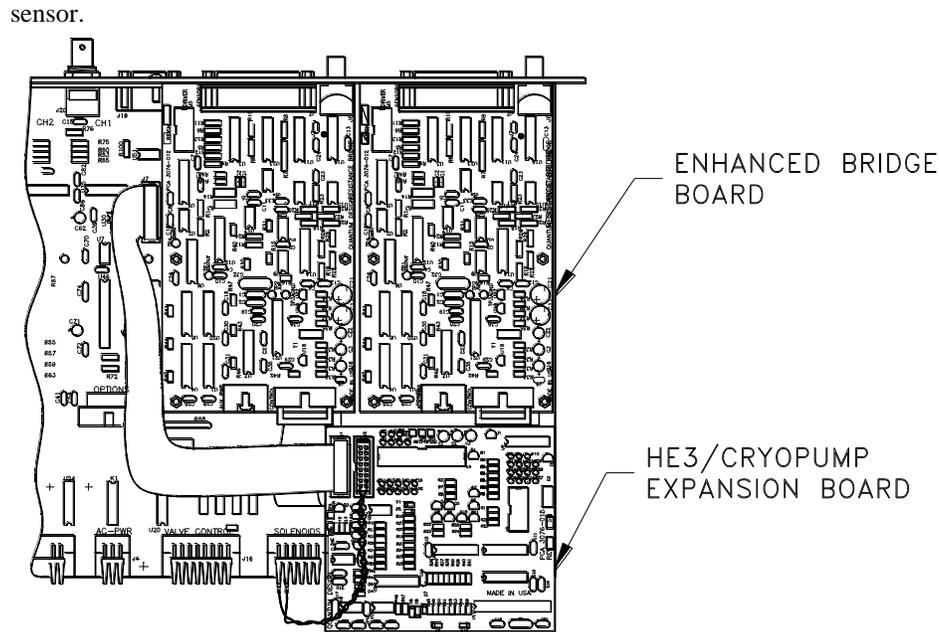


Figure 2-11: Expansion Circuit Board and Enhanced User Bridge Board in Model 6000

2.9 Option-Specific Hardware

2.9.1 Resistivity Measurements

Special eight-pin sample mounts provided with the Helium-3 system are designed for mounting samples for transport measurements that can be performed with the Resistivity option or the AC Transport option.

When the Helium-3 insert is installed and the standard user bridge cable is connected, resistance measurements can be performed on channels 1 and 2 on the bridge board. Refer to the *Physical Property Measurement System: Resistivity Option User's Manual* for information about wiring samples for four-wire resistance measurements to the eight-pin sample mounts and for information about taking resistance measurements when the Helium-3 insert is installed.

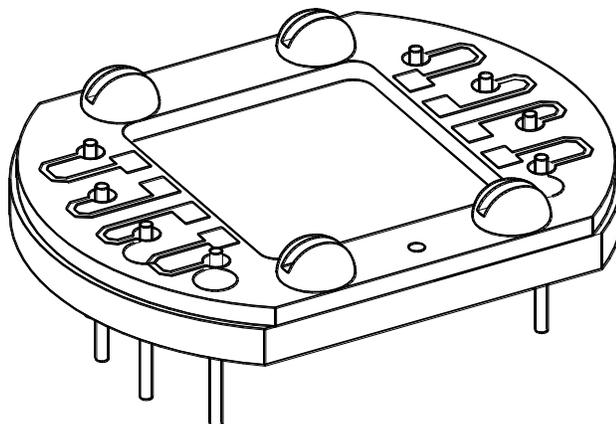


Figure 2-12: Sample Mount for Electrical Transport Measurements

2.9.2 AC Transport Measurements

Special eight-pin sample mounts (see Figure 2-9) provided with the Helium-3 system are designed for mounting samples for transport measurements that can be performed with the Resistivity option or the AC Transport option.

The ACT/Helium-3 probe cable (part number 3084-518) must be connected to take ACT measurements when the Helium-3 insert is installed. This cable splits off the ACT signals from the Helium-3 thermometer. The thermometer signal is plugged into channel 4 of the bridge board.

Refer to the Physical Property Measurement System: AC Transport Option User's Manual for information about wiring samples for ACT measurements to the eight-pin sample mounts and for information about taking ACT measurements when the Helium-3 insert is installed.

2.9.3 Electrical Transport Option Measurement

Special eight-pin sample mounts (see Figure 2-13) provided with the Helium-3 system are designed for mounting samples for transport measurements that can be performed with the Electrical Transport option.

The helium-3 experiment cable (3092-359-01) must be connected to take Electrical transport measurement using ETO option when the Helium-3 insert is installed. This cable splits off the sample channels from the Helium-3 thermometer. The thermometer signal is plugged either into channel 4 of the bridge board for GPIB version or to the Helium-3 control module for CAN version.

Refer to the Physical Property Measurement System: Electrical Transport Option User's Manual for information about the wiring samples for ETO measurements to the eight-pin sample mounts and for information about taking ETO measurements when the Helium-3 insert is installed.

2.9.4 Heat Capacity Measurements

Special Heat Capacity sample mounts have been developed for use with the Helium-3 system. These sample mounts have been designed to fit into the smaller space that is available, and the

thermometer is designed to operate to below 0.4 K. Also, thinner wires are used for the platform support to provide longer sample thermalization times.

A special 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 used for performing the automated calibration procedure on a new Heat Capacity sample mount. A platform stabilizer plug prevents vibration of heat capacity platforms during measurements in a magnetic field.

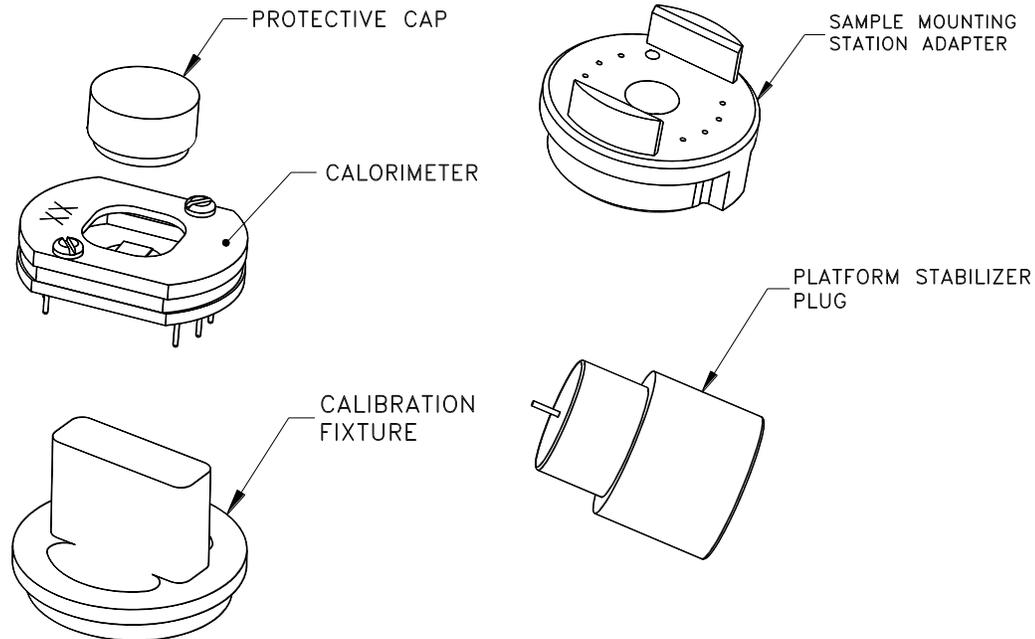


Figure 2-13: Heat Capacity Sample Mount and Accessories

For more information about taking heat capacity measurements when the Helium-3 insert is installed, refer to the *Physical Property Measurement System: Heat Capacity Option User's Manual*.

2.10 Other Accessories

- **Grease.** Apiezon N Grease and Apiezon H Grease are included with the Helium-3 system. Grease is used on the contact fingers near the bottom of the probe for improved thermal contact between the sample chamber and probe. Grease can also be used between the sample mount and sample holder, as well as between the sample mount and sample. N Grease is recommended unless the temperature will exceed 320 K. If the temperature will exceed 320 K, H Grease should be used because it does not melt.
- **Wrenches and screws.** Two small hex wrenches are provided for mounting samples and adjusting the sample holder position. Small screws are provided for attaching both the Resistivity and Heat Capacity sample mounts to the sample holder on the bottom of the insert.
- **Electrical splitter box.** An electrical splitter box (part number 4076-060-01) attaches to the auxiliary port on the back of the Model 6000. The purpose of this is to allow the connections

to be made to the Helium-3 controller through this port, without blocking access to the port for other user functions.

- **Storage accessories.** When the probe is stored in the cart, the probe cover can be placed over the end of the probe, and the vinyl O-ring cover can be placed over the white O-ring.

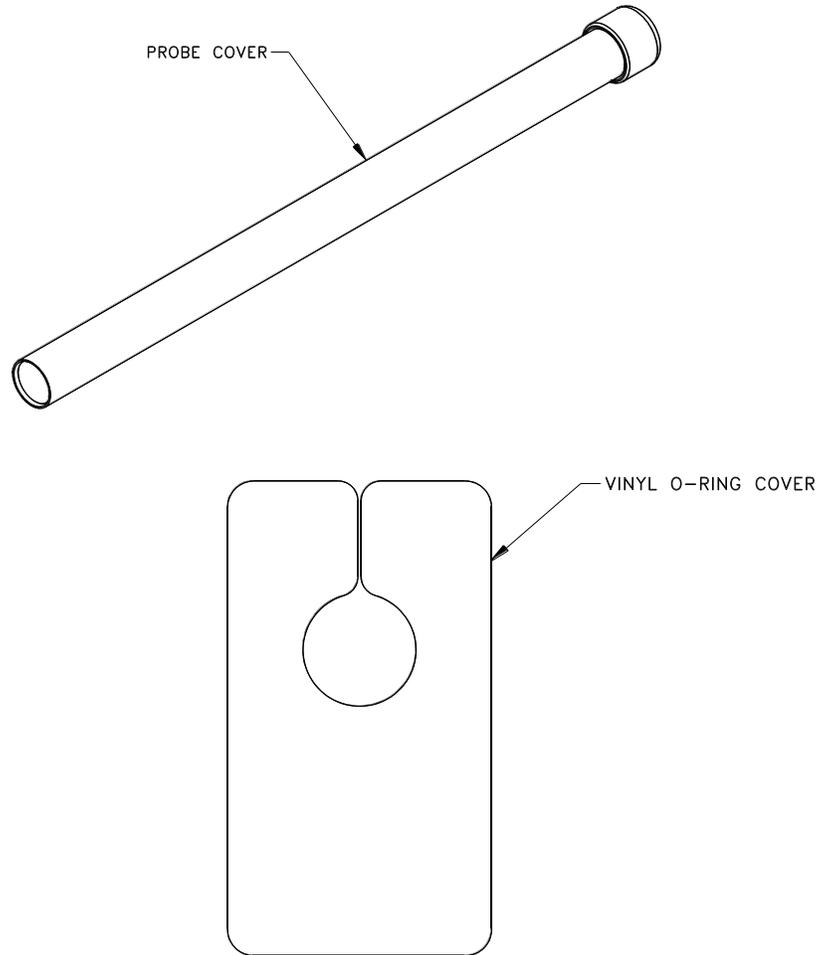


Figure 2-14: Storage Accessories

Software

3.1 Introduction

This chapter contains the following information:

- Section 3.2 discusses the architecture of the Helium-3 software.
- Section 3.3 discusses temperature-control data logging.
- Section 3.4 discusses the Helium-3 maintenance utility.

3.2 Helium-3 Software Architecture

The software provided with the Helium-3 system consists of a main temperature control module, diagnostics utilities, and maintenance utilities. This software is usually installed in the following folder depending on the platform.

1. PPMS: C:\QdPpms\He3Option\
2. DynaCool: C:\QdDynaCool\He3Option\

The configuration information for the specific serial number is contained in the file `Config\He3Option.cfg`, located in the above folder.

To enable temperature control with the Helium-3 insert, the Helium-3 Control Console application must be running on the PC. This application implements the various control modes described in Section 1.4 and controls the transitions between them. All real-time aspects of the temperature control program, including PID control, are executed either by Model 6000 PPMS Controller for GPIB version or by Helium-3 module for CAN version.

3.2.1 Temperature Command Redirection – PPMS Platform

This section describes the temperature command redirection for Helium-3 when installed in a PPMS. For a DynaCool system, refer to the next section.

The Helium-3 system temperature control state engine is executed on the PC. However, the standard interface to the normal PPMS temperature control is handled completely through the

Model 6000. When any application program needs to set a new temperature, it is done by sending a GPIB command to the Model 6000, executing a sequence with temperature commands, or by simply 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 this interface while the main temperature control engine executes on the PC.

When the Helium-3 software is initialized, 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 command redirection is cancelled when the control program is terminated. In this mode, the 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 Helium-3 software is running and the commands are redirected. When temperature control is redirected, a small up-and-down arrow appears by the **TEMP** command. The arrow indicates that temperature control is being processed on the PC. If you use the Model 6000 **Temperature** menu to enter a temperature, an “He3” indicator appears in the upper left corner of the Model 6000 front panel. If you enter a temperature command, the stability indicator on the Model 6000 front panel reads “PC” until the software returns the appropriate stability. 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, the “PC” indicator may remain lit and you do not see the normal status messages. To fix the problem, you must restart the Helium-3 Control Console application. The software will automatically recover from the crash. If you want to recover normal PPMS control without restarting the Helium-3 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 Temperature Command Redirection – DynaCool Platform

This section describes the temperature command redirection for Helium-3 when installed in a DynaCool system.

The Helium-3 system temperature control state engine is executed on the PC. However, the standard interface to the normal temperature control is handled completely through the DynaCool MultiVu software. When a user or any application program needs to set a new temperature, it is done entering the new set point in the Temperature System dialog box, or by executing a sequence with temperature commands. To ensure compatibility with existing software, a special software architecture has been chosen to preserve this interface while the main temperature control engine executes in the Helium-3 software.

When the Helium-3 software is initialized, it sends a command to DynaCool MultiVu that instructs it to forward future temperature commands to the Helium-3 software. The entire DynaCool system then operates in this special redirected mode until command redirection is cancelled when the Helium-3 control program is terminated. In this mode, the temperature control status information is also redirected. The Helium-3 control program determines whether the temperature is “Chasing,” “Near,” or “Stable” and sets the status word in DynaCool MultiVu appropriately. Any sequence running on DynaCool MultiVu can determine the state of the temperature control engine by querying this status word.

3.2.3 Software Initialization and Self-Test

The control software is normally launched just before or after the Helium-3 insert is installed in the PPMS sample chamber. The software functions as a separate program that runs in the background. It can be launched from within PPMS MultiVu or from Windows.

When the software is launched, it reads configuration information from the `He3Option.cfg` file. If the insert is already in the sample chamber, the insert configures the Model 6000 for temperature command redirection, and then the control software enters the standby state. In this state, the temperature control remains inactive until a new temperature set point is entered. At this point, a hardware self-test is performed and the temperature control begins.

If the insert is not in the sample chamber, the system enters the probe-out state. See Figure 3-1. In this state, the software is effectively inactive. Any new temperature set points are simply passed on to the standard PPMS temperature control engine. Only when the insert is installed will the self-test begin.

The self-test can identify a number of hardware failures. However, the primary purpose is to detect cables that are not properly connected.

3.2.4 Helium-3 Option Control Console

The main window of the Helium-3 Control Console application is used to access status information in real time and in a logging mode. You can also shut down the temperature control engine and force it into the shutdown mode when necessary.

The **Status** panel provides a short description of the current state of the Helium-3 temperature control.

The **Logging** button opens the **LoggingDialog**, from which you can monitor and log different hardware parameters related to the option's inner workings.

The **Shut Down He3 Engine** button prompts the Helium-3 option to enter the standby state. In the standby state, the Helium-3 temperature control is idle while it waits for a new set point, and the Helium-3 hardware is shut down.

The **Close** button exits the Helium-3 option. The option's temperature control engine should be shut down before an attempt is made to exit the option. If **Close** is selected and the temperature control engine has not been shut down, a message pops up to remind you to shut down the temperature control engine.

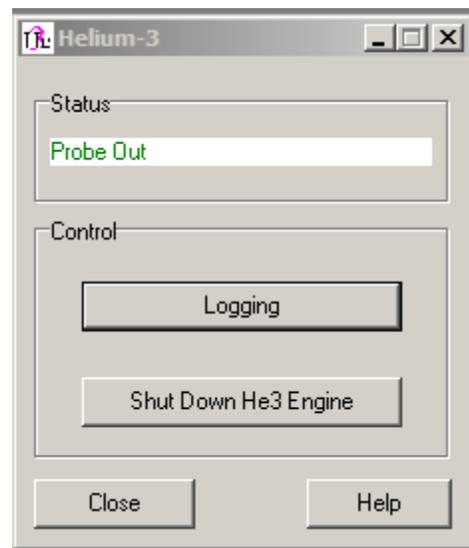


Figure 3-1: Helium-3 Option Control Console

3.3 Temperature-Control Data Logging

Sometimes it may become necessary to monitor the various state variables of the Helium-3 temperature control engine. This is usually required only to diagnose problems with the temperature control.

3.3.1 LoggingDialog

The **LoggingDialog** (refer to Figures 3-2 through 3-4) in the option control console allows you to monitor different diagnostic parameters for the Helium-3 option. It produces files that are compatible with the PPMS MultiVu data file format and can therefore be displayed in real time within PPMS MultiVu. It provides you with considerable information about the option's inner workings.

To use the Logger, you (1) select the file to which the diagnostics will be written, (2) select a logging frequency, (3) choose the fields to log, and then (4) push the **Start** button. The Logger remembers the state it was in the last time the Helium-3 option was running, so it may start logging as soon as the option is launched. The Logger does not remember whether the **Overwrite** box was checked, and by default it always appends data to the selected file.

3.3.2 Logging Set Up

The **General** tab in the **LoggingDialog** allows you to set a file by either browsing over existing files or by entering a new name for a file in the **Diagnostics File Name** text box. It also allows you to select whether to log at a fixed interval or to log just on events.

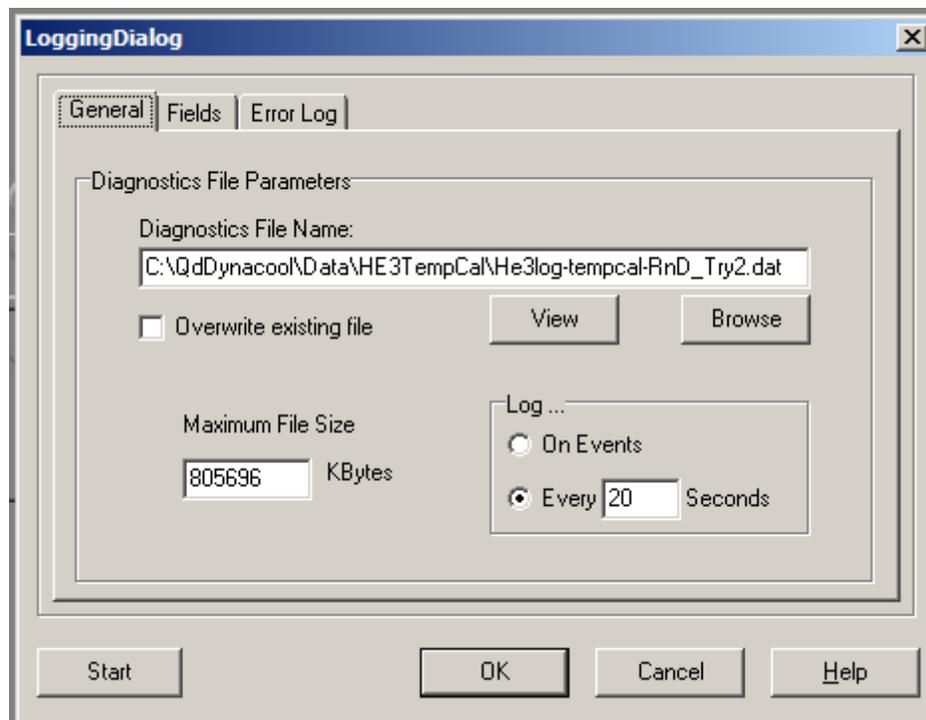


Figure 3-2: **General** Tab in **LoggingDialog**

3.3.3 Selecting the Fields

The **Fields** tab in the **LoggingDialog** allows both the real-time monitoring of the different parameters as well as the selection of parameters for the log file. Parameters that are checked while the logging to file is running go into the log file only after you accept the changes by selecting the **OK** button.

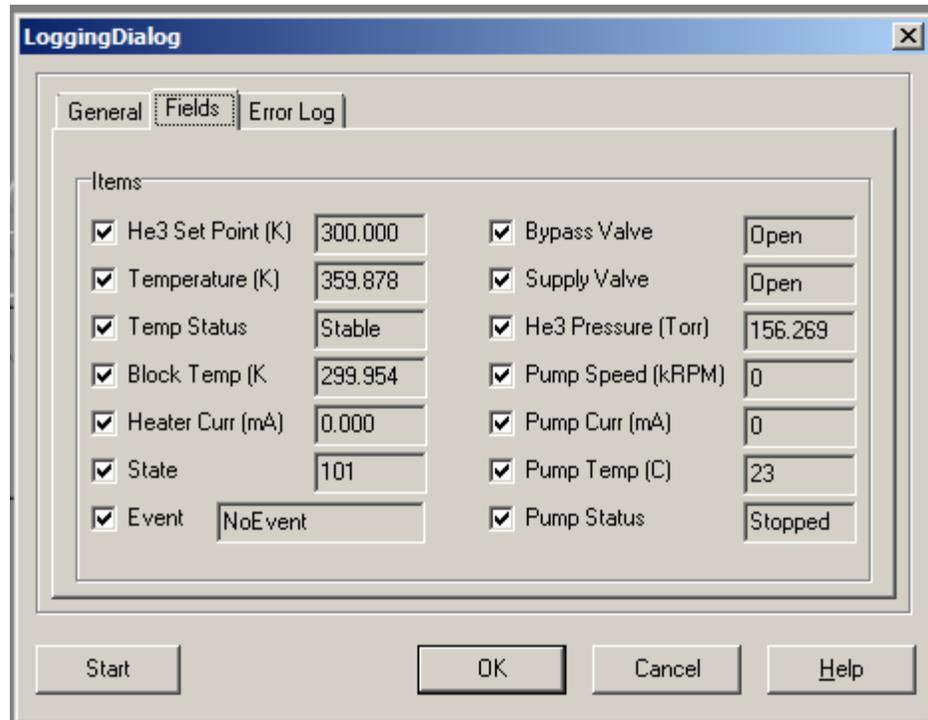


Figure 3-3: **Fields** Tab in **LoggingDialog**

Table 3-1: Helium-3 Log Fields

LOG FIELD	FUNCTION
He3 Set Point	Temperature set point for Helium-3 system. Helium-3 set point is active only at temperatures lower than ≈ 4 K. At higher temperatures, set point reverts to normal PPMS set point.
Temperature	Temperature as reported by Helium-3 system thermometer located close to sample and thus sample temperature reported by PPMS MultiVu and other Quantum Design applications. When Helium-3 system enters standby state, temperature reverts to PPMS thermometry.
Temperature Status	Stability status for sample temperature (for example, “Chasing,” “Near,” “Stable”).
Block Temperature	PPMS, or block, temperature.
Heater Current	Approximate current, in milliamps, through Helium-3 system heater.
State ID	ID number corresponding to actual state of temperature state machine. There is close correspondence between state ID number and status description in Helium-3 option control console.
Event	Brief description of event being processed by state machine.

Bypass Valve	State of bypass valve. A “1” indicates bypass valve is open. This is normally the case when system is in standby state or one-shot state.
Supply Valve	State of supply valve. A “1” indicates supply valve is open. This is normally the case when system is in circulating state.
He3 Pressure	Gas pressure in Helium-3 tank. ³ He gas is completely enclosed in system, so it can be related to liquid level on pot. Therefore a large pressure indicates a large amount of gas in tank and an empty pot, and a small pressure indicates that most ³ He gas has condensed down into pot.
Pump Speed	Speed, in kRPM, of Helium-3 turbo pump. Value is generally between 9 and 75 kRPM.
Pump Current	Turbo pump drive current, in mA. Pump has power-limiting features that hold current below certain value. This value depends on current operating speed and gas flow. Generally, it is limited to be less than about 600.
Pump Temperature	Temperature of turbo pump bearing. Pump has temperature-limiting feature that causes it to shut down if its temperature goes above 60°C.
Pump Status	Brief description of status of turbo pump controller.

3.3.4 Error Log

The **Error Log** tab in the **LoggingDialog** displays all the error, warning, and information messages that the Helium-3 option has produced since it was launched. The tab also displays the software version number.

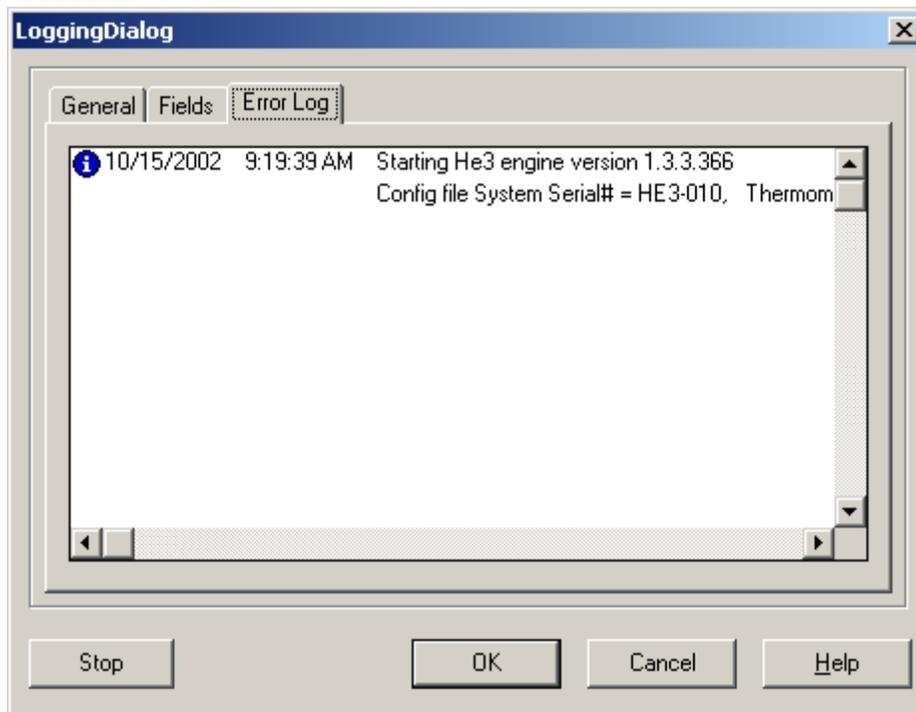


Figure 3-4: Error Log Tab in LoggingDialog

3.4 Helium-3 Maintenance Utility (He3GasMon.exe)

The Helium-3 Option Maintenance Utility application (He3GasMon) has been designed to provide direct manipulation of the Helium-3 system hardware, as well as pre-written macros (wizards) for performing certain common maintenance procedures. The location of the utility is `\qdp\ppms\He3Option\He3GasMon.exe`. To run the maintenance procedure, you select the Windows **Start** button and then you select **Programs >> Quantum Design >> He3Option >> He3GasMon**.

CAUTION!

You are advised to close the Helium-3 Control Console application before you start the He3GasMon maintenance utility. You are strongly advised to close the Helium-3 Control Console application before you run any of the software wizards. During normal operation, the Helium-3 system is designed to be controlled by only the Helium-3 Control Console application.

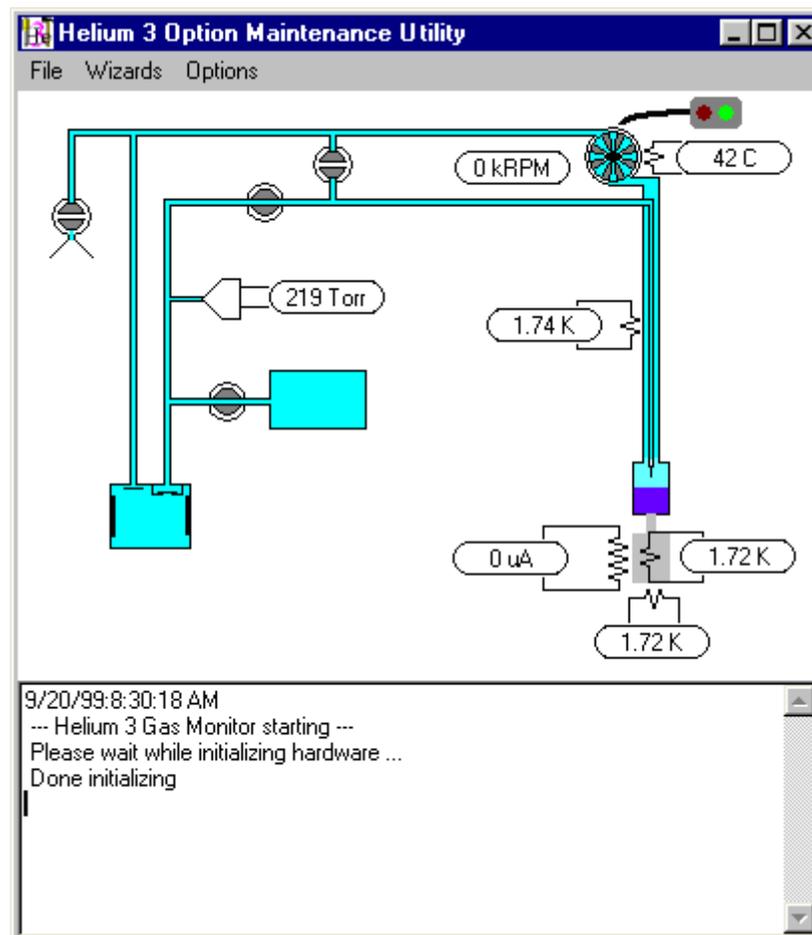


Figure 3-5: Helium-3 Maintenance Utility

3.4.1 Visual Manipulation of the Gas-Handling System

The maintenance utility provides a visual representation of the Helium-3 gas-handling system. When the program is activated, it is in the display-only mode. To allow control of the hardware, you check various options under the **Options** menu. For example, if you check the **Allow Valve Control** item, you can double-click on a valve to toggle its state. If the **Allow Pump Control** item is checked, you may activate the turbo pump by clicking on the icon representing the pump switch.

3.4.2 Wizards

The **Wizards** menu in the maintenance utility contains a number of macros for performing a variety of maintenance procedures. Specific instructions for these wizards are in chapter 6.

A log file containing all messages from the wizards is located in the file `\qdppms\He3Option\He3GasMonWizards.log`.

System Operation with PPMS

4.1 Introduction

This chapter contains the following information:

- Section 4.2 explains how to set up and shut down the Helium-3 system.
- Section 4.3 summarizes procedures for taking measurements when the Helium-3 system is installed.
- Section 4.4 explains how to manually position the sample holder area.

4.2 System Setup and Shutdown

Note: After the Helium-3 option has been set up for the first time, very little work is required to shut down and reactivate the option between usages. The electrical connections between the cart, computer, and Model 6000 PPMS Controller are easily disconnected and reconnected. Software initialization is handled automatically by simply launching the Helium-3 Control Console application.

The following instructions assume that the Helium-3 cart with the Helium-3 insert and the backing pump has been previously disconnected from the cabinet and computer 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 cart adjacent to the PPMS probe and rack.

4.2.1 Connecting the Electrical Cables

4.2.1.1 HELIUM-3 GPIB VERSION FOR PPMS PLATFORM

Using the Figure 4-1 for guidance, complete the electrical connections from the Helium-3 system GPIB version by following the steps below.

1. Position the Helium-3 cart so that it is near the PPMS dewar, control computer, and PPMS electronics cabinet.

2. Note whether your system has a single black connector for all control signals. If your system has a single black connector, you may reconnect the cable to the cart. Otherwise, follow steps 3 and 4.
3. Plug the 25-pin connector into the “P8–Auxiliary” port on the back of the Model 6000.
4. Plug the serial cable into the “COM1” port on the back of the computer.
5. Plug the power connector into the power strip on the back of the electronics cabinet.
6. Plug in the He-3 experiment cable to the gray Lemo on the probe, this cable splits the He-3 thermometer to a 4 pin Lemo and user measurement channels (Channel 1 and Channel 2) to a 14 pin Lemo receptacle.
7. Plug in the gray Lemo cable for your measurement option to He-3 user experiment cable box. Refer to the appropriate option manual for specific information about the Lemo cable that enables your option.

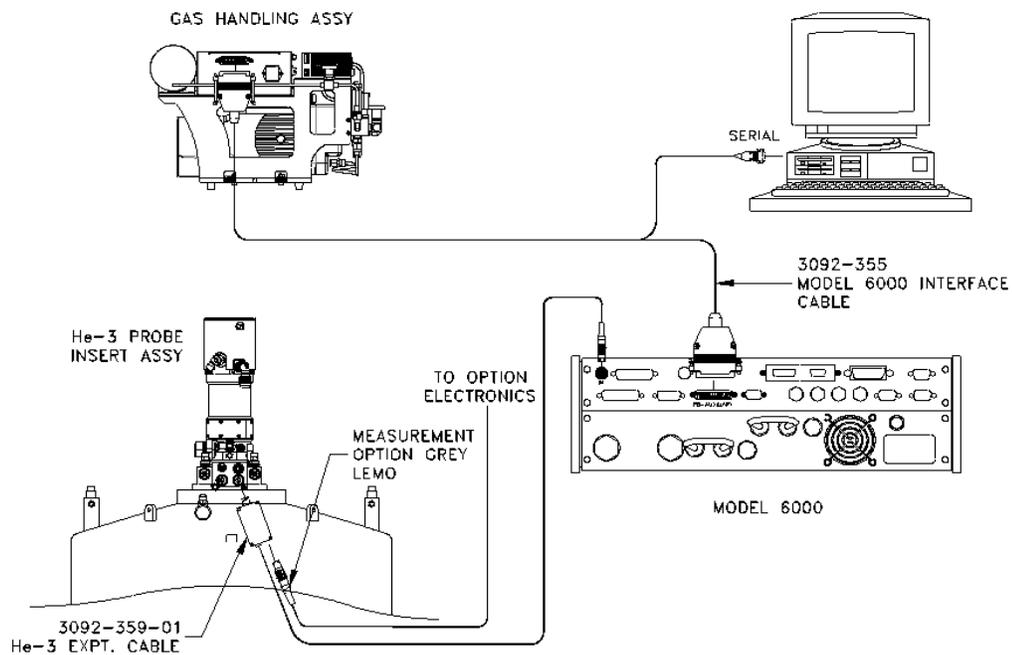


Figure 4-1: He-3 option GPIB version connection diagram on a PPMS

4.2.1.2 HELIUM-3 CAN VERSION FOR PPMS PLATFORM

Using the Figure 4-2 for guidance, complete the electrical connections from the Helium-3 system CAN version by following the steps below.

1. Position the Helium-3 cart so that it is near the PPMS dewar, control computer, and PPMS electronics cabinet.
2. Connect He-3 CAN interface cable to Gas Handling System and HE-3 CAN module on Model 1000 CAN tower. Refer to Tables B-2 and B-3 in Appendix B and Figure 4-2.
3. Connect the CAN cable to Model 1000 tower and USB-CAN dongle.
4. Connect the USB-CAN dongle to computer.

5. Connect the power connector of the tower to the power strip in the electronic cabinet.
6. Plug the power connector into the power strip on the front of the electronics cabinet.
7. Plug in the He-3 experiment cable to the gray Lemo on the probe, this cable splits the He-3 thermometer to a 4 pin Lemo and user measurement channels (Channel 1 and Channel 2) to a 14 pin Lemo receptacle.
8. Plug in the gray Lemo cable for your measurement option to He-3 user experiment cable box. Refer to the appropriate option manual for specific information about the Lemo cable that enables your option.

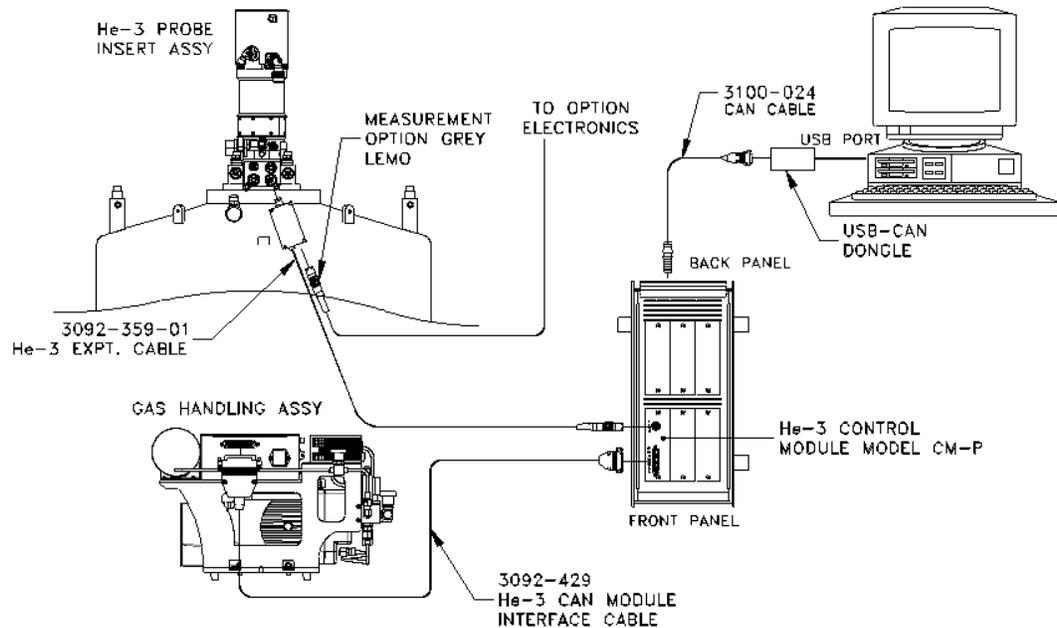


Figure 4-2: He-3 option CAN version connection diagram on PPMS platform

4.2.2 Launching the Helium-3 Control Console Application

Once the hardware is connected, you may activate the software. The Helium-3 Control Console application is a stand-alone program. As such, it can be invoked by launching the executable from the Windows desktop. It may also be accessed through the PPMS MultiVu **Utilities**►**Activate Option** menu option, where you click on **Helium3** and then select the **Activate** button.

Notice that you can minimize the Helium-3 option control console. The Helium-3 software runs in the background, so the option control console does not have to be visible in the PPMS MultiVu interface.

Launching the Helium-3 option starts up the Helium-3 engine. When the option is activated, it defaults to the standby state. As soon as a new temperature set point is received, the software wakes up, runs a series of hardware tests, and starts controlling.

4.2.3 Shutting Down the Helium-3 Control Console Application

When you will no longer use the Helium-3 system, set it in standby state by selecting the **Shut Down He3 Engine** button in the Helium-3 option control console (see Figure 3-1). Selecting this button makes the Helium-3 option relinquish temperature control on the current set point to the Model 6000. However, if the Helium-3 insert is still in the sample chamber and a new temperature set point is selected, the Helium-3 option takes over temperature control again.

After the system enters the standby state, you can quit the application by selecting the **Close** button in the Helium-3 option control console.

If for some reason the software terminates abnormally or crashes, always restart the application and shut it down properly. When the software is shut down normally, it properly shuts down the hardware and places it in a safe state.

4.2.4 Disconnecting and Storing the Hardware

Follow the instructions in Section 2 for removing and safely storing the Helium-3 insert on the cart. Then simply reverse the steps in Section 4.1.1 to disconnect and store the cart in a safe place. Quick Secure the Helium-3 gas if you plan to store the option for a period more than two months. Run the "Quick Secure Helium-3 Gas" wizard to secure the gas (Refer to section 6.3.5)

CAUTION!

Failure to secure the gas before a long term storage may result in loss of Helium-3 gas.

4.3 Taking Measurements

The following procedures are generic and assume that all electrical connections to the Helium-3 system have already been made. For specific information about using a PPMS measurement option with the Helium-3 system, refer to the appropriate option manual.

4.3.1 Start the Software

1. Activate the Helium-3 Control Console application as described in Section 4.2.2.
2. Start up the software application for your measurement option (Resistivity, Electrical Transport, or Heat Capacity). You can (a) select the program icon or (b) select **Utilities >> Activate Option** in PPMS MultiVu, click on the option name, and then select the **Activate** button.

4.3.2 Attach the Sample Mount

1. Follow the sample preparation and mounting instructions as contained in the specific user manual for the type of measurement you will perform.
2. Apply a small amount of Apiezon H Grease or N Grease to the bottom side of the plug-in sample mount. Applying additional grease may be unnecessary if grease is left over from a previous run.
3. Place the Helium-3 insert in the Helium-3 cart if the insert is not already in the cart. The insert and cart are specifically designed so that when the insert is in the cart, the sample holder is at a convenient height for you to reach.
4. Hold the plug-in sample mount so that the guide hole on it lines up with the guide pin on the sample holder and then push the sample mount onto the holder. If necessary, use tweezers or a similar tool to push the sample mount into position.
5. Use a hex ball driver to gently snug—but not tighten—the two hex Allen screws on the back of the sample holder. Be careful to hold the ball driver as straight as possible so that the screw heads aren't stripped. It is not possible to hold the driver perfectly straight on systems with the horizontal sample mount, so be especially careful when tightening the screws on such a system. Snug the screws until they just hold the plug-in sample mount against the mounting plate.
6. Apply a small amount of Apiezon H Grease or N Grease to the condenser contact fingers that are on the gold-plated sample cage area on the insert. Refer to Figure 4-1. Apply only enough grease so that the grease is not visible, but the contact fingers just feel tacky.

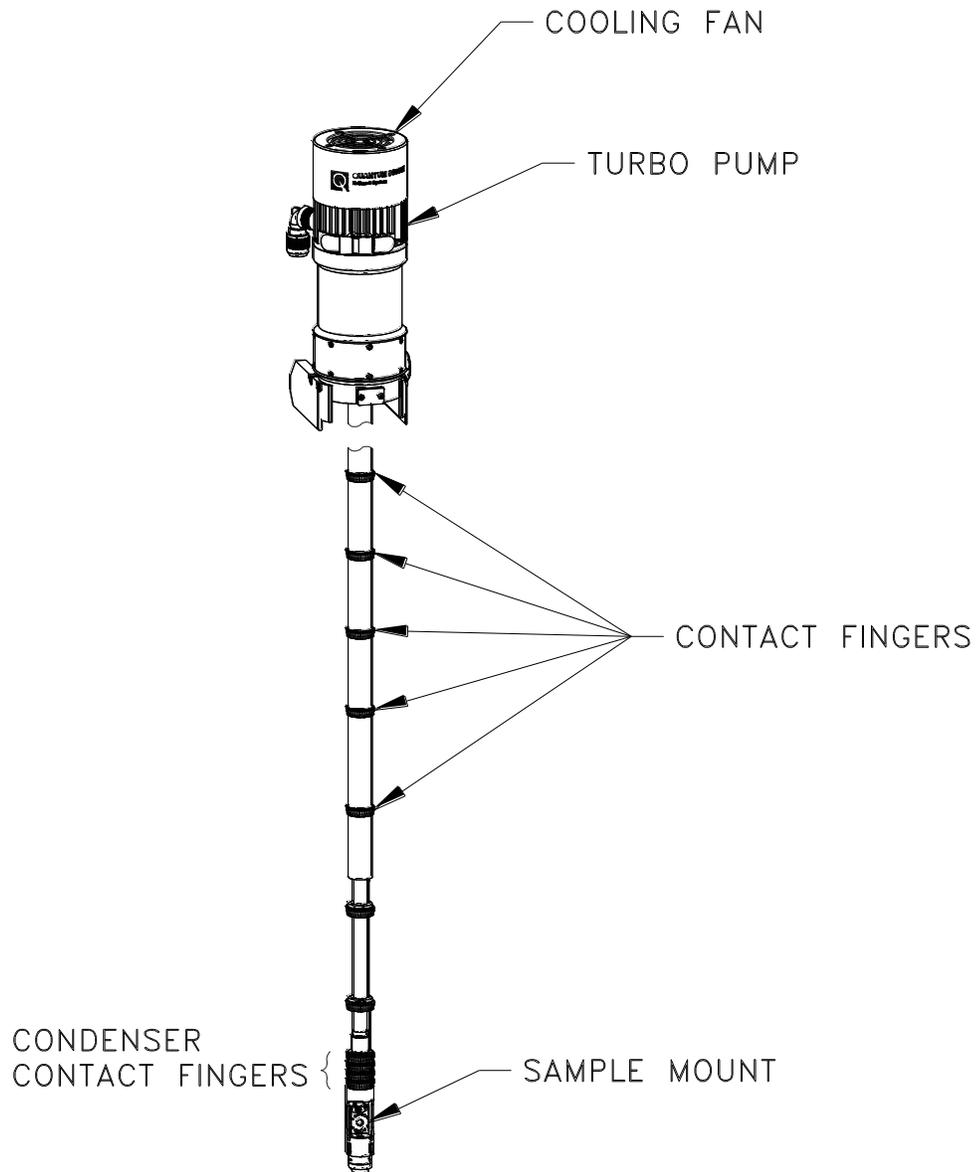


Figure 4-3: Condenser Contact Fingers

4.3.3 Approximate Available Space for User Experiments

In the HE-3 using horizontal sample mount, and assuming the electro-transport puck is mounted to the sample mount, there will be 20mm diameter (avoid i.d. of the tube frame of HE-3 fridge) with 14.5mm height. Removing the sample mount increases the available height to ~24mm, but the user will have to provide thermometer and heater for temp control.

4.3.4 Install the Helium-3 Insert

CAUTION!

Handle the Helium-3 insert very carefully. To help prevent the refrigerator probe on the insert from being damaged, stand on the provided step stool when you install the insert into the PPMS sample chamber or when you remove it.

Note: If the Helium-3 insert has been recently removed from the PPMS sample chamber, verify that the refrigerator probe is dry. The shaft of the probe is usually cold when it is removed, and any moisture that condenses on the probe can impede the high-vacuum system.

1. Place the step stool that is included with the Helium-3 option next to the PPMS dewar.
2. Start up the installation wizard that will step you through the procedures to run a sample measurement for your measurement option. The installation wizard prompts the system to set the temperature to room temperature and flood the sample chamber with helium.
3. Wait for the installation wizard to indicate that system conditions are correctly set for installing the Helium-3 insert in the sample chamber.
4. Unlatch and remove the clamp that surrounds the sample chamber access port. If a flange is in the access port, remove the flange. Refer to the appropriate option manual for more specific information about opening the sample chamber access port.
5. Open the safety latch on the Helium-3 cart, and then lift the Helium-3 insert out of the cart.
6. Inspect the white silicon O-ring that is at the base of the turbo pump. It may be necessary to clean it and apply a small amount of Apiezon M Grease to ensure that it provides a good sealing surface.
7. Turn the Helium-3 insert so that the turbo pump is at the top of the probe shaft and the sample cage area is at the bottom of the shaft. Refer to Figure 2-1.
8. Verify that the cables attached to the turbo pump lie naturally and are not overly twisted or kinked. Straighten the cables, if necessary. The cables can be damaged if you insert the refrigerator probe into the sample chamber while the cables are twisted or kinked.
9. Step up on the stool, and lift the insert above the dewar. Hold the insert so that the fins on the turbo pump face backwards and the large, black mesh cable hangs over the right side of the insert.

CAUTION!

Avoid stressing the shaft of the refrigerator probe in any way while you are installing the Helium-3 insert in the sample chamber. The material out of which the shaft is constructed is thin and fragile and can easily bend or dent.

10. Slowly lower the insert into the PPMS sample chamber while securely holding the turbo pump. You should feel the resistance of the contact fingers increase in the last 4–5 cm.
11. Make sure the O-ring is properly seated on the sample chamber flange.
12. Complete any additional tasks the installation wizard prompts you to perform before you run the measurement.

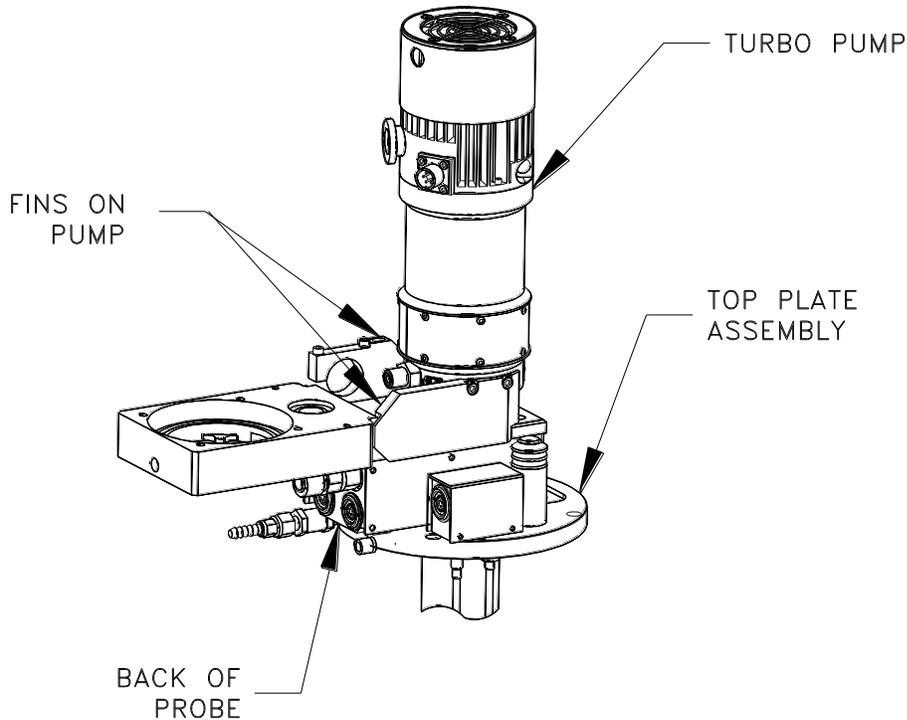


Figure 4-4: View of Turbo Pump When Helium-3 Insert Is Installed in Sample Chamber

4.3.5 Run the Measurement

1. Verify that the sample chamber is purged. The measurement option should have prompted you to do this. If you did not run the installation wizard, you must purge the chamber before you run the measurement.
2. Run a measurement as you would normally. All procedures for running a measurement in immediate mode or sequence mode are identical whether or not the Helium-3 insert is installed. However, when the insert is installed you can take measurements below 0.4 K.

4.3.6 Remove the Helium-3 Insert

1. Complete any additional tasks the installation wizard prompts you to perform.
2. Wait for the installation wizard to indicate that system conditions are correctly set for removing the insert from the sample chamber.

Note: The system temperature must be between 295–315 K and the sample chamber must be flooded with helium before you can remove the insert from the sample chamber. The installation wizard correctly sets these system conditions. You must set the temperature and vent the chamber if you did not run the installation wizard.

CAUTION!

Avoid stressing the shaft of the refrigerator probe in any way while you are removing the Helium-3 insert in the sample chamber. The material out of which the shaft is constructed is thin and fragile and can easily bend or dent.

3. Step up on the stool, and then gently pull the insert out of the dewar.
4. Turn the insert so that the turbo pump is at the bottom of the probe shaft and the sample cage area is at the top of the shaft. Place the insert in the cart, then close the safety latch on the cart.

4.3.7 Remove the Sample Mount

1. Use a hex wrench to loosen—but not remove—the two hex Allen screws on the back of the sample holder. Keep the two screws inside the slots on the sample holder. When the screws remain in the slots, you can use them to gently push the plug-in sample mount away from the socket on the probe.
2. Remove the sample mount by pulling it straight out using fingers or tweezers. The pins can be easily bent, so be careful.

4.4 Manually Positioning the Sample Holder Area

If your Helium-3 system has a vertical sample holder, you can manually position the sample holder so that it is aligned at different angles. This adjustment allows you to vary the angle between the plate of the sample holder and the applied magnetic field in systems with a transverse magnet. The sample holder can be manually rotated up to 140° around the longitudinal axis of the sample space.

The orientation of the sample holder is set using two hex Allen screws. Small wrenches have been provided for loosening these screws. When adjusting the orientation, make sure the heater wire attached to the underside of the mounting plate is not stressed. Tighten the screws snugly enough that you get good thermal contact between the part that rotates and the part that is stationary. Be careful that you do not strip the screws; they are very small.

System Operation with DynaCool

5.1 Introduction

This chapter contains the following information:

- Section 5.2 explains how to set up and shut down the Helium-3 system.
- Section 5.3 summarizes procedures for taking measurements when the Helium-3 system is installed.
- Section 5.4 explains how to manually position the sample holder area.

5.2 System Setup and Shutdown

Note: After the Helium-3 option has been set up for the first time, very little work is required to shut down and reactivate the option between usages. The electrical connections between the cart, computer, and Helium-3 CAN Controller are easily disconnected and reconnected. Software initialization is handled automatically by simply launching the Helium-3 Control Console application.

The following instructions assume that the Helium-3 cart with the Helium-3 insert and the backing pump has been previously disconnected from the cabinet and computer 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 cart adjacent to the PPMS probe and rack.

5.2.1 Connecting the Electrical Cables

1. Position the Helium-3 cart so that it is near the PPMS dewar, control computer, and PPMS electronics cabinet.
2. Connect the He-3 CAN interface cable (25-pin connector) to the Helium-3 CAN Module on the Model 1000 CAN tower and the Gas Handling System. Refer to Tables B-2 and B-3 in Appendix B and Figure 5-1.
3. Connect the CAN cable to Model 1000 tower and USB-CAN dongle.
4. Connect the USB-CAN dongle to computer.

5. Connect the power connector of the tower into the power strip on the back on the electronics cabinet.
6. Plug the power connector into the power outlet on the front of the electronics cabinet.
7. Plug in the He-3 experiment cable to the gray Lemo on the probe. This cable splits the He-3 thermometer to a 4-pin Lemo and user measurement channels (Channel 1 and Channel 2) to a 14-pin Lemo receptacle.
8. Plug in the gray Lemo cable for your measurement option. Refer to the appropriate option manual for specific information about the Lemo cable that enables your option.

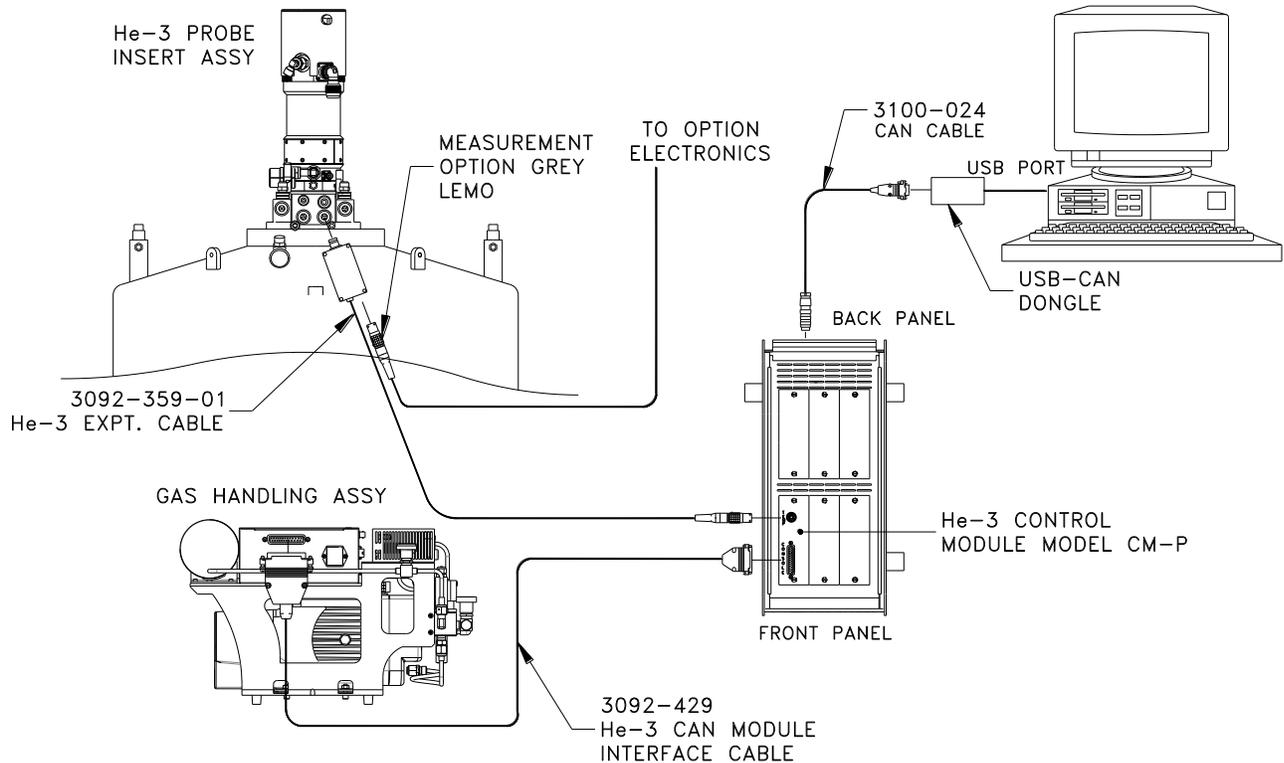


Figure 5-1: He-3 option CAN version connection diagram on PPMS platform

5.2.2 Launching the Helium-3 Control Console Application

Once the hardware is connected, you may activate the software. The Helium-3 Control Console application is a stand-alone program. As such, it can be invoked by launching the executable from the Windows desktop. It may also be accessed through the PPMS MultiVu **Utilities** ➤ **Activate Option** menu option, where you click on **Helium3** and then select the **Activate** button.

Notice that you can minimize the Helium-3 option control console. The Helium-3 software runs in the background, so the option control console does not have to be visible in the PPMS MultiVu interface.

Launching the Helium-3 option automatically starts up the Helium-3 engine. When the option is activated, it defaults to the standby state. As soon as a new temperature set point is received, the software wakes up, runs a series of hardware tests, and starts controlling.

5.2.3 Shutting Down the Helium-3 Control Console Application

When you will no longer use the Helium-3 system, put it into the standby state by selecting the **Shut Down He3 Engine** button in the Helium-3 option control console (see Figure 3-1). Selecting this button makes the Helium-3 option relinquish temperature control on the current set point to the CAN module. However, if the Helium-3 insert is still in the sample chamber and a new temperature set point is selected, the Helium-3 option takes over temperature control again.

After the system enters the standby state, you can quit the application by selecting the **Close** button in the Helium-3 option control console.

If for some reason the software terminates abnormally or crashes, always restart the application and shut it down properly. When the software is shut down normally, it properly shuts down the hardware and places it in a safe state.

5.2.4 Disconnecting and Storing the Hardware

Follow the instructions in Section 2 for removing and safely storing the Helium-3 insert on the cart. Then simply reverse the steps in Section 5.2.1 to disconnect and store the cart in a safe place. If you plan to store the option for a period longer than two months secure the ^3He gas. Run the "Quick Secure Helium-3 Gas" wizard to secure the gas (Refer to section 6.3.5)

CAUTION!

Failure to secure the gas before a long term storage may result in loss of Helium-3 gas.

5.3 Taking Measurements

The following procedures are generic and assume that all electrical connections to the Helium-3 system have already been made. For specific information about using a PPMS measurement option with the Helium-3 system, refer to the appropriate option manual.

5.3.1 Start the Software

1. Activate the Helium-3 Control Console application as described in Section 5.2.2.
2. Start up the software application for your measurement option (Resistivity, Electrical Transport, or Heat Capacity). You can (a) select the program icon or (b) select **Utilities >> Activate Option** in PPMS MultiVu. Click on the option name, and then select the **Activate** button.

5.3.2 Attach the Sample Mount

1. Follow the sample preparation and mounting instructions as contained in the specific user manual for the type of measurement you will perform.
2. Apply a small amount of Apiezon H Grease or N Grease to the bottom side of the plug-in sample mount. Applying additional grease may be unnecessary if grease is left over from a previous run.
3. Place the Helium-3 insert in the Helium-3 cart if the insert is not already in the cart. The insert and cart are specifically designed so that when the insert is in the cart, the sample holder is at a convenient height for you to reach.
4. Hold the plug-in sample mount so that the guide hole on it lines up with the guide pin on the sample holder and then push the sample mount onto the holder. If necessary, use tweezers or a similar tool to push the sample mount into position.
5. Use a hex ball driver to gently snug—but not tighten—the two hex Allen screws on the back of the sample holder. Be careful to hold the ball driver as straight as possible so that the screw heads aren't stripped. It is not possible to hold the driver perfectly straight on systems with the horizontal sample mount, so be especially careful when tightening the screws on such a system. Snug the screws until they just hold the plug-in sample mount against the mounting plate.
6. Apply a small amount of Apiezon H Grease or N Grease to the condenser contact fingers that are on the gold-plated sample cage area on the insert. Refer to Figure 5-2. Apply only enough grease so that the grease is not visible, but the contact fingers just feel tacky.

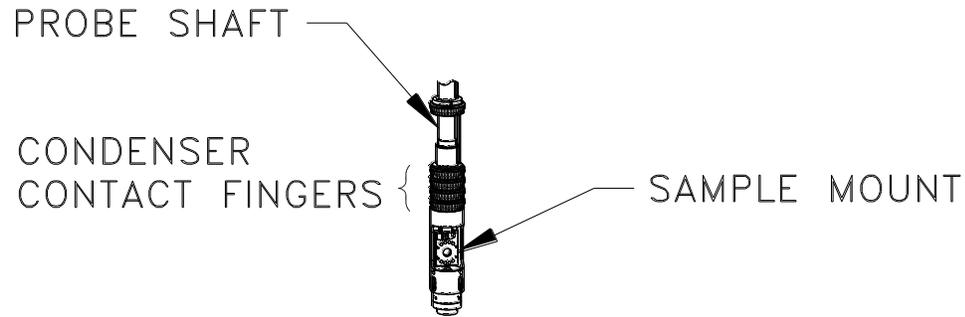


Figure 5-2: Condenser Contact Fingers

5.3.3 Install the Helium-3 Insert

CAUTION!

Handle the Helium-3 insert very carefully. To help prevent the refrigerator probe on the insert from being damaged, stand on the provided step stool when you install the insert into the PPMS sample chamber or when you remove it.

Note: If the Helium-3 insert has been recently removed from the PPMS sample chamber, verify that the refrigerator probe is dry. The shaft of the probe is usually cold when it is removed, and any moisture that condenses on the probe can impede the high-vacuum system.

1. Place the step stool that is included with the Helium-3 option next to the PPMS dewar.
2. Start up the installation wizard that will step you through the procedures to run a sample measurement for your measurement option. The installation wizard prompts the system to set the temperature to room temperature and flood the sample chamber with helium.
3. Wait for the installation wizard to indicate that system conditions are correctly set for installing the Helium-3 insert in the sample chamber.
4. Unlatch and remove the clamp that surrounds the sample chamber access port. If a flange is in the access port, remove the flange. Refer to the appropriate option manual for more specific information about opening the sample chamber access port.
5. Open the safety latch on the Helium-3 cart, and then lift the Helium-3 insert out of the cart.
6. Inspect the white silicon O-ring that is at the base of the turbo pump. It may be necessary to clean it and apply a small amount of Apiezon M Grease to ensure that it provides a good sealing surface.
7. Turn the Helium-3 insert so that the turbo pump is at the top of the probe shaft and the sample cage area is at the bottom of the shaft. Refer to Figure 2-1.
8. Verify that the cables attached to the turbo pump lie naturally and are not overly twisted or kinked. Straighten the cables, if necessary. The cables can be damaged if you insert the refrigerator probe into the sample chamber while the cables are twisted or kinked.
9. Step up on the stool, and lift the insert above the dewar. Hold the insert so that the fins on the turbo pump face backwards and the large, black mesh cable hangs over the right side of the insert.

CAUTION!

Avoid stressing the shaft of the refrigerator probe in any way while you are installing the Helium-3 insert in the sample chamber. The material out of which the shaft is constructed is thin and fragile and can easily bend or dent.

10. Slowly lower the insert into the PPMS sample chamber while securely holding the turbo pump. You should feel the resistance of the contact fingers increase in the last 4–5 cm.
11. Make sure the O-ring is properly seated on the sample chamber flange.
12. Complete any additional tasks the installation wizard prompts you to perform before you run the measurement.

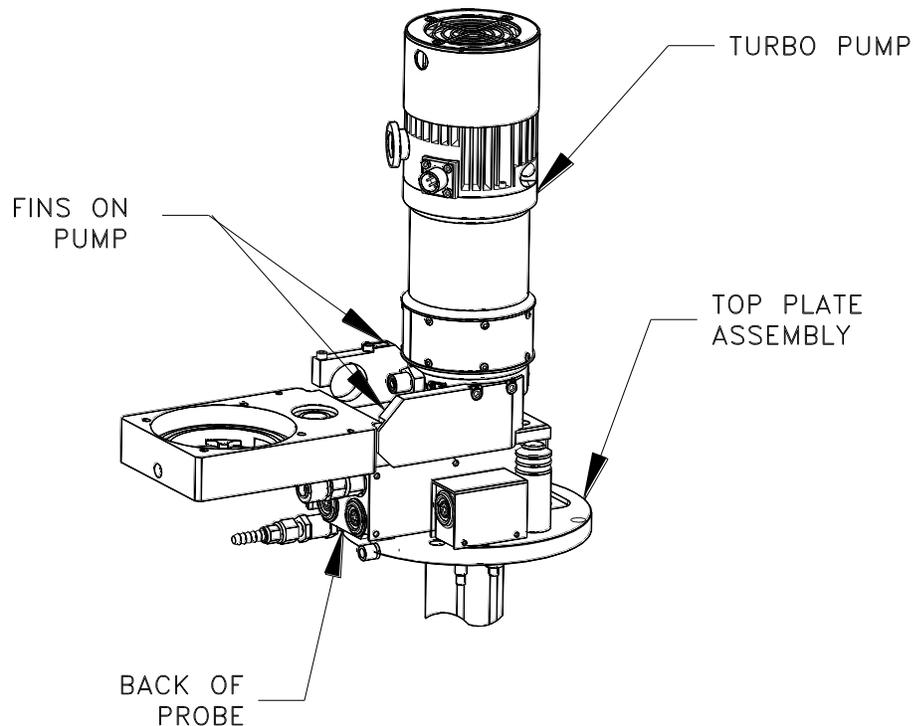


Figure 5-3: View of Turbo Pump When Helium-3 Insert Is Installed in Sample Chamber

5.3.4 Run the Measurement

1. Verify that the sample chamber is purged. The measurement option should have prompted you to do this. If you did not run the installation wizard, you must purge the chamber before you run the measurement.
2. Run a measurement as you would normally. All procedures for running a measurement in immediate mode or sequence mode are identical whether or not the Helium-3 insert is installed. However, when the insert is installed you can take measurements below 0.4 K.

5.3.5 Remove the Helium-3 Insert

1. Complete any additional tasks the installation wizard prompts you to perform.
2. Wait for the installation wizard to indicate that system conditions are correctly set for removing the insert from the sample chamber.

Note: The system temperature must be between 295–315 K, and the sample chamber must be flooded with helium before you can remove the insert from the sample chamber. The installation wizard correctly sets these system conditions. You must set the temperature and vent the chamber if you did not run the installation wizard.

CAUTION!

Avoid stressing the shaft of the refrigerator probe in any way while you are removing the Helium-3 insert in the sample chamber. The material out of which the shaft is constructed is thin and fragile and can easily bend or dent.

3. Step up on the stool, and then gently pull the insert out of the dewar.
4. Turn the insert so that the turbo pump is at the bottom of the probe shaft and the sample cage area is at the top of the shaft. Place the insert in the cart, then close the safety latch on the cart

5.3.6 Remove the Sample Mount

1. Use a hex wrench to loosen—but not remove—the two hex Allen screws on the back of the sample holder. Keep the two screws inside the slots on the sample holder. When the screws remain in the slots, you can use them to gently push the plug-in sample mount away from the socket on the probe.
2. Remove the sample mount by pulling it straight out using fingers or tweezers. The pins can be easily bent, so be careful.

5.4 Manually Positioning the Sample Holder Area

If your Helium-3 system has a vertical sample holder, you can manually position the sample holder so that it is aligned at different angles. This adjustment allows you to vary the angle between the plate of the sample holder and the applied magnetic field in systems with a transverse magnet. The sample holder can be manually rotated up to 140° around the longitudinal axis of the sample space.

The orientation of the sample holder is set using two hex Allen screws. Small wrenches have been provided for loosening these screws. When adjusting the orientation, make sure the heater wire attached to the underside of the mounting plate is not stressed. Tighten the screws snugly enough that you get good thermal contact between the part that rotates and the part that is stationary. Be careful that you do not strip the screws; they are very small.

Troubleshooting and Maintenance

6.1 Introduction

This chapter contains the following information:

- Section 6.2 is a troubleshooting guide for the Helium-3 system.
- Section 6.3 explains how to perform common maintenance procedures for the Helium-3 system.

6.2 Troubleshooting Guide

The following sections describe the causes, symptoms, and remedies for the most common problems that can occur while using the Helium-3 system.

6.2.1 Plugged Impedance

A plugged impedance is the most common reason the system fails to cool below 1.8 K.

CAUSE	The impedance that feeds ^3He gas into the pot of the Helium-3 system becomes plugged by a gradual buildup of contamination from out-gassing and minute leaks. The ice plug stops the circulation of gas.
SYMPTOM	The temperature control operates normally but cannot cool below the normal PPMS base temperature of about 1.8 K. On the first attempt to go below about 1.8 K, the Helium-3 Control Console application indicates the system is in the “He3 Circulating” state, but does not reach or stabilize at a lower temperature.
VERIFICATION	<p>If the system has not been cryocleaned in more than a month or so, the lack of cleaning is the most likely cause of the problem. Do the following to verify the problem:</p> <ol style="list-style-type: none"> 1. Shut down the Helium-3 Control Console application while the system is still at the lowest temperature it can reach. 2. Wait about 10 minutes or until the PPMS base temperature is less than 2 K. 3. Enter a temperature of 0.5 K from the front panel of the Model 6000 or via PPMS MultiVu.

4. Wait for the Helium-3 pump to start running.
5. Open the **LoggingDialog** in the Helium-3 option control console, and select the **Fields** tab.
6. Note the He3 pressure, He3 temperature, and block temperature.

While the system was shut down, ^3He gas was allowed to accumulate; hence the He3 temperature may drop well below 1.8 K as ^3He gas evaporates from the pot. However, if the impedance is plugged, the pot eventually goes dry, and the temperature rises back up to the block temperature. Also, if the impedance is plugged, the pressure raises continuously to some value greater than about 250 torr, reaching a maximum value when the pot goes dry. The pressure never decreases if the impedance is plugged. In addition, the turbo pump speed probably increases to the maximum of 75 kRPMs once the pot is dry, because there is little gas flow in this case.

REMEDY Follow the cryocleaning procedure in Section 6.3.1 to remove the contamination from the ^3He gas.

6.2.2 Poor Thermal Contact to Condenser

CAUSE This problem is caused by weak mechanical contact between the lower walls of the sample chamber and the contact fingers located along the shaft of the Helium-3 insert. Although problems are usually caused by poor contact with the condenser contact fingers at the very bottom, poor contact higher up the shaft can lead to poor performance as well.

SYMPTOM In the worst case, the system does not cool below about 2 K. In moderate cases, it may take more than 1 hour to cool from 1.5 to 0.5 K. Unlike the symptoms of a plugged impedance, the system may be able to maintain temperatures below 1.5 K.

VERIFICATION The following procedures detail the simplest way to determine whether the contact is sufficient:

1. Press the **Shut Down He3 Engine** button and close the console if it is currently running, and then launch the He3GasMon utility as described in Section 3.4.
2. Run the “**Check Condenser Contact**” wizard.

This procedure makes certain the sample chamber is at high vacuum and that no condensed ^3He is present in the pot. It then sets the temperature to the lowest value and measures any temperature difference between the ^3He thermometer and the PPMS system thermometer.

Generally the wizard reports a temperature difference of about 0.1 K between the PPMS neck temperature and helium-3 thermometer.

REMEDY If the temperature difference is larger than about 0.15 K, you should make sure the contact fingers are not completely dry. Add a small amount of Apiezon N or H Grease as necessary. In unusual cases, the bottom of the sample chamber may have been previously stretched by an ice plug resulting from a gross leak into the sample chamber, thus requiring servicing by Quantum Design.

6.2.3 Gas System Leaks

CAUSE	Gas system leaks can result from damage to the probe, corrosion, or from a rupture of a diaphragm in the pumping assembly.
SYMPTOM	If there is a small system leak, the system may simply plug more frequently than normal and hence require cryocleaning more often. The system is considered normal if it takes a month or more to develop a plug without cryo-cleaning. In more extreme cases, the Helium-3 application may report a loss of pressure. Refer to Section 6.2.1, “Plugged Impedance,” to verify the symptoms of lack of cryocleaning.
VERIFICATION-REMEDY	<p>To test for a gross leak and a large loss of ^3He, run the “Pressure Verification” wizard as indicated in Section 6.3.7. If the pressure is more than 10% different, there is probably a leak. To perform a complete leak check of the system, you need to have a helium leak detector. If you are prepared to perform a complete leak check, run the following wizards:</p> <ol style="list-style-type: none">1. “Clean and Secure He3 Gas” wizard (Section 6.3.5).2. “Leak Check System” wizard (Section 6.3.8). If leaks are found, the system must be repaired. If the system is leaking around the heads of the backing pump, it may be necessary to replace the rubber diaphragms (Section 6.3.9). After all repairs are made, rerun the wizard and leak check the system until no leaks are found.3. Run the “Evacuate and Seal” wizard (Section 6.3.6) once the system is leak free.

6.2.4 Backing Pump Deterioration

CAUSE	The constant motion of the rubber components in the backing pump causes the rubber to gradually break down over time. The diaphragms may crack and leak or the flapper valves may stop sealing properly.
SYMPTOM	Cracked diaphragms produce a gas system leak as described above in Section 6.2.3. If the flapper valves degrade, the system does not leak or plug, but may exhibit poor cooling characteristics due to poor backing pressure for the turbo pump. The system may no longer be able to cool below 0.4 K when in one-shot mode.
VERIFICATION	To determine whether the flapper valves in the backing pump need servicing, run the “Pressure Verification” wizard as described in Section 6.3.7. If the wizard indicates that the turbo pump cannot achieve the maximum speed, the flapper valves may require servicing.
REMEDY	<p>To replace the diaphragms or service the flapper valves, perform the following procedures:</p> <ol style="list-style-type: none">1. Run the “Clean and Secure He3 Gas” wizard (Section 6.3.5) to save the gas into the tank.2. Complete the steps (Section 6.3.9) to replace the diaphragms or service the flapper valves.3. Run the “Leak Check System” wizard (Section 6.3.8) to leak check the pump.4. Run the “Evacuate and Seal” wizard (Section 6.3.6).

6.2.5 Software and Firmware Problems

Due to the complexity of software and firmware, it is sometimes necessary to reinitialize the entire system. Although symptoms vary, many intermittent software problems that appear over time are “fixed” by the following procedure:

6.2.5.1 HELIUM-3 GPIB VERSION

1. Exit all programs - in an orderly fashion if possible - executing on the PC.
2. Shut down the PC from the Windows **Start** menu in the normal way.
3. Turn off the Model 6000 PPMS Controller and wait for at least 1 minute. Do not cycle the power more rapidly than this because there is a risk of damaging the controller.
4. If you have a Model 6500 Option Controller, turn it off as well.
5. Turn the Model 6000 and the Model 6500 back on and restart the computer.

6.2.5.2 HELIUM -3 CAN VERSION

1. Exit all programs - in an orderly fashion if possible - executing on the PC.
2. Shut down the PC from the Windows **Start** menu in the normal way.
3. Turn off the CAN tower and wait for at least 1 minute. Do not cycle the power more rapidly than this because there is a risk of damaging the controller.
4. Turn the CAN tower back on and restart the computer.

6.3 Maintenance Procedures and Wizards

Most of the maintenance procedures described in this section are at least partially automated by using the Helium-3 Option Maintenance Utility application (He3GasMon). The maintenance utility provides a graphic display of the gas-handling system (see Figure 3-5) along with a menu of software wizards that guide you through the most common maintenance procedures. Each wizard mentioned in the sections below consists of a series of partially automated instructions for completing specific maintenance procedures.

To run the maintenance utility, you must first shut down and quit the Helium-3 Control Console application if it is already running. Then from the Windows **Start** menu, select **Programs >> Quantum Design >> He3Option >> He3GasMon**. To launch a specific wizard, select it from the **Wizards** menu.

CAUTION!

You are advised to close the **Helium-3 Control Console** application before you start the **He3GasMon** maintenance utility. You are strongly advised to close the **Helium-3 Control Console** application before you run any of the software wizards. During normal operation, the Helium-3 system is designed to be controlled by only the **Helium-3 Control Console** application.

Several of the procedures require the use of the cryocleaning pump-out hose (part number 4092-630) provided with the system. This hose connects between the PPMS sample chamber and the Helium-3 cart and uses the PPMS as a high-vacuum pumping station.

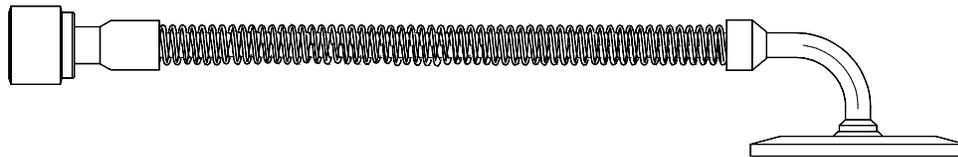


Figure 6-1: Cryocleaning Hose

6.3.1 Cryocleaning the Helium-3 Gas Using LN₂ Cold Trap

If the Helium-3 impedance becomes plugged, it will be necessary to cryoclean the ³He gas. A plugged impedance results in a lack of cooling below about 1.8 K. To prevent impedance plugs, perform the cryocleaning procedure about once every two weeks when the system is under heavy usage. Or perform the procedure before using the Helium-3 system if it has been idle for more than two weeks. Little-to-no ³He gas is lost during this procedure, so you can perform the operation often without the expense of adding ³He gas to the system.

To perform this operation, run the “**Clean He3 with LN₂ Trap**” wizard. The software will guide you through the following steps:

1. Verify that the LN₂ trap is *not* in an overpressure condition. If it is, regenerate the trap by using the “Regenerate LN₂ Trap” wizard. See Section 6.3.2 for more information.
2. Cool the LN₂ cold trap by submerging it in liquid nitrogen.
3. Apply a thin film of Apiezon M grease to the double-sealing connectors on the cold trap and on the Helium-3 gas manifold.
4. Use the double-sealing connectors to connect the cold trap to the gas manifold. When prompted, slowly open the valve labeled, “CRYOCLEAN” located on the gas handling system.
5. Wait while the wizard flushes the ³He gas into and out of the cold trap, removing all non-helium gases.
6. Slowly close the “CRYOCLEAN” valve and disconnect the cold trap from the Helium-3 gas manifold. Wipe off any excess grease on the surfaces of the double-sealing connectors and replace the rubber caps to prevent dirt from getting on the connectors.
7. The wizard will automatically measure the pressure of the ³He gas in the system and update the configuration file.

The entire operation will take about an hour if there is a fair amount of contaminant gas present and less time if the ³He gas is clean.

6.3.2 Regenerating the 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 in 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 cryocleaning hose.
2. Pump 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.

6.3.3 Cryocleaning the Helium-3 Gas Using PPMS

This procedure allows cryocleaning of the ³He gas without the LN₂ cold trap. Instead of removing the contaminants using the cold trap, the Helium-3 probe is cooled in the PPMS sample chamber, and contaminants are condensed on the walls of the Helium-3 probe. Only perform this if you do not have the LN₂ cold trap.

To perform this operation, run the **“Cryo-Clean He3”** wizard. The software guides you through the following steps:

1. Cool the Helium-3 insert to about 10 K to allow the cold surfaces inside the insert to freeze out the non-helium contamination.
2. Pump the remaining ³He gas into the reservoir tank and close the tank valve.
3. Warm up the system and place the Helium-3 insert in the Helium-3 cart.
4. Remove contaminants from the system by evacuating the entire gas-handling system except for the tank where the clean ³He gas is stored.
5. Seal the system and let the ³He gas back into the system.
6. Measure the remaining pressure of the ³He gas in the system and update the configuration file.

The entire cryocleaning procedure requires from 3 to 10 hours. The length of the procedure depends on whether the probe is already cold when you start the procedure, and how long you choose to pump on the system when it is warm.

Notice that each time this procedure is performed, the system loses about 3% of the remaining ³He gas. Hence, this procedure may be performed about 10 times before it is necessary to add more gas. See Section 6.3.4 for adding ³He gas to the system.

6.3.4 Adding Helium-3 Gas to the System

After running the cryocleaning procedure multiple times, or after repeated maintenance operations, it will become necessary to add more ³He gas to the system.

To perform this procedure you need a bottle of ³He gas connected to a tee pump-out fixture. This fixture consists of a tee joint with the following three branches. The first branch consists of a female VCO-type, face-seal fitting for connecting to the **“FILL”** port on the backing pump assembly. The

second branch consists of a valve (labeled “Gas Valve”) connected to the ^3He gas supply bottle. The third branch consists of a valve (labeled “Pump”) connected to a male VCO-type, face-seal fitting. The fittings, without the ^3He gas, are provided with the Helium-3 system. See Figure 6-2.

To obtain the gas, please contact Quantum Design or a gas supplier directly. The 99.8% pure grade is adequate for the Helium-3 system. This can be obtained from Cambridge Isotope Laboratories, Inc., (www.isotope.com) under the part number HE-29. To be able to connect to the fill manifold, the bottle must have a male $\frac{1}{4}$ NPT-type fitting. It is also recommended that the bottle be fitted with a needle valve for precise control of filling.

Before starting the procedure, verify that the system is in the normal operating configuration with the “TANK” valve open and the “FILL” valve closed. Then run the “Add He3 to system” wizard. The software guides you through the following steps:

1. Place the Helium-3 insert in the cart.
2. Connect the fill-bottle fixture to the fill port on the backing pump assembly and evacuate the fixture to remove any air from the fill port.
3. Add ^3He gas to the system until the pressure reaches about 250 torr for Diaphragm pump version gas handling system and 150 torr for Scroll pump version gas handling system.

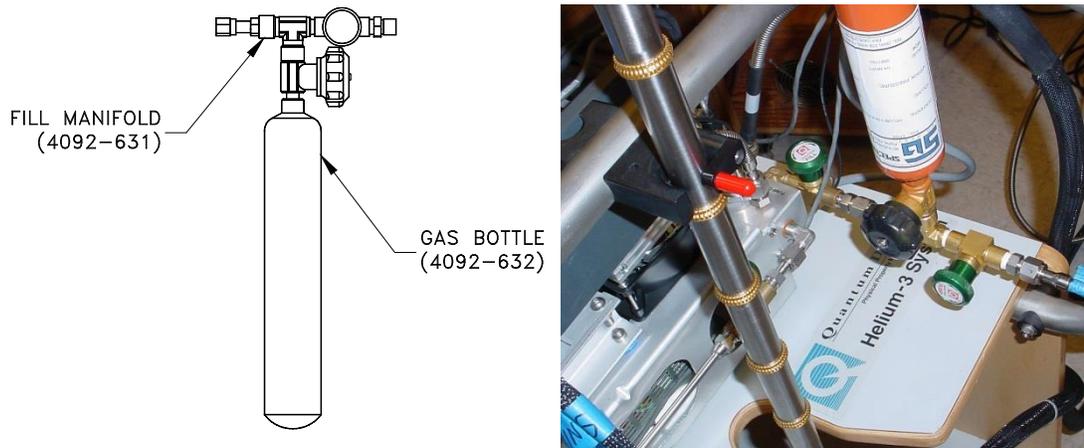


Figure 6-2: (Left Pic) Helium Fill Manifold (Right Pic) Helium Fill Manifold Connected to HE-3 GHS

6.3.5 Securing the Helium-3 Gas in the Tank

In order to save as much of the ^3He gas as possible before performing any maintenance operation that requires opening the system, you must secure the ^3He gas into the tank. Maintenance operations requiring that the system be opened include repairing a bad system component like a solenoid valve, or repairing or cleaning the backing pump.

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

To save the ^3He gas into the tank, run the “Clean and Secure He3 Gas” wizard. The software guides you through the following steps:

1. Cool the Helium-3 insert to about 10 K to allow the cold surfaces inside the insert to freeze out the non-helium contamination.
2. Pump the remaining ^3He gas into the reservoir tank and close the manual valve.
3. Warm up the system and place the Helium-3 insert into the Helium-3 cart.

If you need to open the system but do not need to clean the gas, you may run the “**Quick Secure He3 Gas**” wizard. This simply performs the second step noted above without the need to thermally cycle the system.

6.3.6 Evacuating and Sealing the System

After the system has been opened for maintenance or cryocleaning, it must be evacuated and the gas stored in the tank must be let back into the system.

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

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

1. Evacuate the system through the “FILL” line.
2. Close the “FILL” valve.
3. Open the “TANK” valve to let the ^3He gas back into the system.
4. Measure the remaining quantity of ^3He gas in the system and update the configuration file.

6.3.7 Verifying the Helium-3 Pressure

This procedure detects the presence of a gross leak in to or out of the ^3He gas system. It also indicates whether the backing pump is providing adequate backing pressure for the turbo pump.

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

Run the “**Pressure Verification**” wizard. The software guides you through the following steps:

1. Run the diaphragm assembly to pump the ^3He gas into the tank.
2. Measure the pressure of the gas in the tank and compare it to the value last recorded in the configuration file.
3. Measure the maximum turbo pump speed. If it is less than 75 kRPM, a warning is issued.

The program indicates whether there is a significant difference between the current pressure and the original pressure. If there is a significant difference, you need to determine whether there is a leak in the system. Leak checking the system requires the use of helium leak detector and someone trained in leak-detector operation. Refer to Section 6.3.8 for more on leak checking.

If this procedure indicates that the turbo pump cannot achieve the maximum speed, the flapper valves in the backing pump require servicing. Refer to Section 6.3.9 for more on rebuilding the backing pump.

6.3.8 Leak Checking the System

Leak checking the ^3He gas space requires the use of 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” valve and the “FILL” valve closed. That is, you must have already run the “Clean and Secure He3 Gas” wizard as described in Section 6.3.5.

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 backing pump assembly. You may want to use the provided cryocleaning hose (part number 4092-630) 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. Run the turbo pump and backing pump while leak checking the system.
5. Once leak checking is complete, close the “FILL” valve and turn off the pump.

If you discover that the system is leaking, the system must be repaired and the above steps repeated until a leak-free system is achieved.

6.3.9 Rebuilding the Backing Pump

This procedure must be followed to service the backing 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 backing pump assembly located inside the cart and the insert will interfere if it is in the cart.
2. Remove the backing 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 backing 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. Measure the maximum height of the center of the diaphragm relative to the pump head surface. The center of the diaphragm should reach a height of 0.380 +/-0.005 inches above the pump head surface.

Caution: If the diaphragm extends higher than this the pump head will be damaged and need to be replaced. 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. Install either (a) the connecting bar or (b) the bellows hose on the top of the backing 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.

6.4 He-3 Status Codes Explanation

Temp status (code)

The temperature status code in the He3 logfile follows the same rules as the main PPMS temperature control (first 4 bits of PPMS status):

- 0 - status unknown
- 1 - stable at target T
- 2 - stable
- 5 - near
- 6 - chasing
- 10 - standby mode

"state (code)" value in the He3 log file (He3Option.dat), e.g. 0.102.200.300.400 .

Each of the numbers in the code represents a state. States evolve from other states, and the last one is the current state.

- 0 : Result := 'Starting up';
- 100 : Result := 'Checking Hardware';
- 101 : Result := 'Initialize Hardware';
- 102 : Result := 'Controlling';
- 103 : Result := 'StandBy';
- 104 : Result := 'Probe Out';
- 105 : Result := 'PPMS StandBy';
- 106 : Result := 'Helium3 StandBy';
- 107 : Result := 'Spinning down turbopump';
- 200 : Result := 'Selecting the temperature range';
- 300 : Result := 'Starting the high temperature SM';
- 301 : Result := 'Going to the high temperarute SM';
- 302 : Result := 'Going to the low temperature SM';
- 303 : Result := 'Starting the low temperature SM';

400 : Result := 'Controlling temperature (fast)';
401 : Result := 'Controlling temperature (slow)';
402 : Result := 'Waiting for temp. for high vacuum';
403 : Result := 'Starting high vacuum';
404 : Result := 'Waiting to achieve low temp.';
405 : Result := 'Controlling temperature';
406 : Result := 'Going to the High temp. SM';
500 : Result := 'Temp. control using He3 heater';
501 : Result := 'Going to system heater control';
502 : Result := 'Going to He3 heater control';
503 : Result := 'Temp. control using system heater';
600 : Result := 'Circulating He3'; // PID parameters changed
601 : Result := 'Using He4 cooling'; // PID parameters changed
602 : Result := 'Stopping He3 circulation'; // pot operation
603 : Result := 'Starting He3 circulation';
604 : Result := 'Evacuating He3 Pot';
605 : Result := 'One shot He3 mode'; // PID parameters changed

Installation Instructions

A.1 Introduction

This appendix contains the following information:

- Section A.2 explains how to install the Helium-3 option in Physical Property Measurement System.

A.2 Installing the Helium-3 Option

These installation instructions assume that the High-Vacuum option (Model P640) is already installed in the PPMS unit. The High-Vacuum option must be installed before you can install the Helium-3 option. Refer to your high-vacuum option manual.

You must have the following tools to install the Helium-3 option:

- Computer running a current version of the DynaCool MultiVu software. Current version can be found at www.qdusa.com
- 9/64-inch hex wrench
- 3/16-inch nut driver
- 9/32-inch nut driver
- Philips and standard screwdrivers

A.2.1 Install the Software

To install the software, follow the printed instructions that are provided with the software disks. If no separate printed instructions are included, perform the following steps:

1. Install PPMS MultiVu. Do the following: (a) insert PPMS MultiVu Software CD into the PC, (b) select the D: drive, (c) run `setup.exe` from the installation CD, and then (d) follow the on-screen instructions.

2. Install the Helium-3 option software. Do the following: (a) insert Helium-3 Installation Software into the PC, (b) select the D: drive, (c) run `setup.exe` from the installation disk, and then (d) follow the on-screen instructions. Copy the configuration file to the HE3 Option folder.
3. The Helium-3 software consists of the installation disk(s), which contain the program and utilities, and a disk containing the configuration files for the Helium-3 insert.
4. Install the Heat Capacity software, version 2.0 beta or later, if you will be using the Heat Capacity option (Model P650). Refer to the procedures in step 2.
5. Install the Electrical Transport Option software, version 1.0.75 or later, if you will be using the Electrical Transport Option (Model P605). Refer to procedure in step 2.

A.2.2 Change the EPROMs



1. Back up the ROM configuration information by running the ROM configuration utility. Do the following: (a) double-click on the **PPMS 32-bit Tools** icon 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 then (f) select **OK**.
2. Turn off 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 the furthest to the left.
7. Install the two new EPROMs 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 notches in the sockets.

A.2.3 Install the Boards

1. Examine the board in the “P1–User Bridge” slot on the Model 6000. If the board does *not* have part number 3076-050, or if there is no board in the slot, install the user bridge board included with the Helium-3 option, and connect the ribbon cable.
After you connect the ribbon cable, tuck it under the user bridge board in order to make room for the expansion circuit board.
2. Remove any connectors plugged into the “P8–Auxiliary” port, and then attach the aluminum splitter box (part number 4076-060-01) to the “P8–Auxiliary” port. Reattach the connectors you just removed.
Note: The remainder of the procedures in this section explain how to install the expansion circuit board (part number 3076-015). If the expansion circuit board is already installed in the lower right corner of the Model 6000, skip to Section A.2.4. A PPMS using the cryopump version of the High-Vacuum option should already include the expansion board.
3. Use a 9/32-inch nut driver to remove the CPU board from the Model 6000. Removing the CPU board exposes a large circuit board, called the motherboard.
4. Check for an installed chip in the U55 socket in the motherboard. If no chip is present, proceed to step 5. If a chip is present in the socket, its label should read “PPMS_SEL 2.” If the revision

and the “CKSUM” numbers are identical to those on the upgrade chip,¹ then you do not need to replace the chip that’s already installed. If the revision or the “CKSUM” numbers are different, replace the chip in the motherboard with the upgrade chip.

5. Insert the PAL chip labeled “PPMS_SEL2” into the empty “U55” socket on the exposed circuit board. The notch on the PAL chip should line up with the notch in the socket.
6. Reinstall the CPU board.
7. Remove any board that is installed in the “P3–Option” slot on the Model 6000.
8. Install the expansion circuit board (part number 3076-015) as follows: (a) remove the nut and washer holding the lower right corner of the motherboard, (b) attach the standoff included with the Helium-3 option, (c) install the expansion board into the standoff, and (d) reinstall the washer and nut.

If the standoff does not stand vertically, you may need to bend it to make it straight. The expansion circuit board will not fit unless the standoff is straight. The two connectors on the underside of the board should completely engage the connectors on the motherboard.

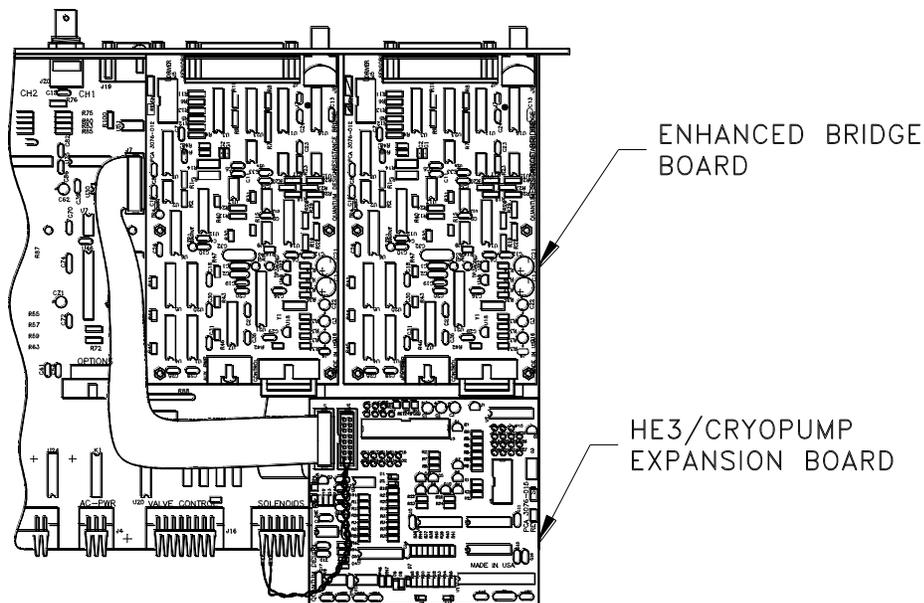


Figure A-1: Expansion Circuit Board and Enhanced User Bridge Board in Model 6000

8. Remove the 16-pin connector at the “J7-Aux-Spare” position on the motherboard and plug it into the “J2” connector on the expansion circuit board.
A jumper connector is installed between the “J24-Solenoids” and “J7-Aux-Spare” positions on the motherboard only if the High-Vacuum option is already installed. The High-Vacuum option must be installed before you can install the Helium-3 option. For installation instructions, refer to your high-vacuum option manual.
9. Install the “Auxspare-to-Expansion Board” ribbon cable (part number 3076-018) between the “J1” connector on the expansion board and the “J7-Aux-Spare” connector on the motherboard. Do not let the ribbon cable touch the large resistors above the “Valve Control” connector; these resistors can get very hot. Try running the cable under the system bridge board.
10. Reinstall the board you removed from the “P3–Option” slot.

¹ Included with the Helium-3 option.

Note: Perform the remainder of the procedures in this section only if the PPMS uses a turbo pump version of the High-Vacuum system. If the PPMS does not include a turbo pump, skip to Section A.2.4.

11. Remove the dongle connector labeled “P8–Auxiliary” from the “P8–Auxiliary” port on the Model 6000. This connector is a rectangular metal box with 25-pin connectors on each end.
12. Place the “P6–Dewar” sticker included with the Helium-3 option over the original sticker on the dongle connector.
13. Unscrew the connector from the “P6–Dewar” port on the Model 6000.
14. Plug the relabeled “P6–Dewar” dongle connector into the “P6–Dewar” port by inserting the dongle between the plug and the jack.

A.2.4 Power Up the Model 6000

1. Put the cover back on top of the Model 6000.
2. Reinsert the Model 6000 power cord.
3. Turn on the power to the Model 6000.
4. Run the ROM configuration utility, and send the previously backed-up ROM configuration to the Model 6000.
5. 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 Helium-3 installation.

A.2.5 Place the Diaphragm Pump Assembly in the Cart

CAUTION!

It is strongly recommended that at least two people handle the pump and probe assembly while completing the following procedure. The Helium-3 refrigerator probe is very fragile. The thin wall of the probe shaft can be easily dented or bent, and the turbo pump mounted on the shaft is very sensitive to mechanical shocks. The diaphragm pump assembly contains many gas-tight seals that could be compromised if the diaphragm pump is dropped or handled roughly.

1. Remove the diaphragm pump assembly and the attached Helium-3 insert from the packing crate.
2. Verify that the power selector on the diaphragm pump assembly is set to the proper line voltage (for example, 110 V or 220 V). To set the voltage, remove the fuse holder and change the selector. The fuse holder is part of the power connect located on the tank end of the controller.
3. Lay the Helium-3 insert on the floor or have someone hold it.
4. Carefully lift up the diaphragm pump assembly. Reattach any electrical connectors that were unplugged for shipping.
5. Carefully place the diaphragm pump assembly at the back of the open cabinet area in the Helium-3 cart. Position the tank so that its four rubber feet fit into the four holes in the floor of the cabinet. Refer to Figure A-2 on the following page. Work with extreme care to avoid torquing, crimping, or loosening any of the attached hoses.

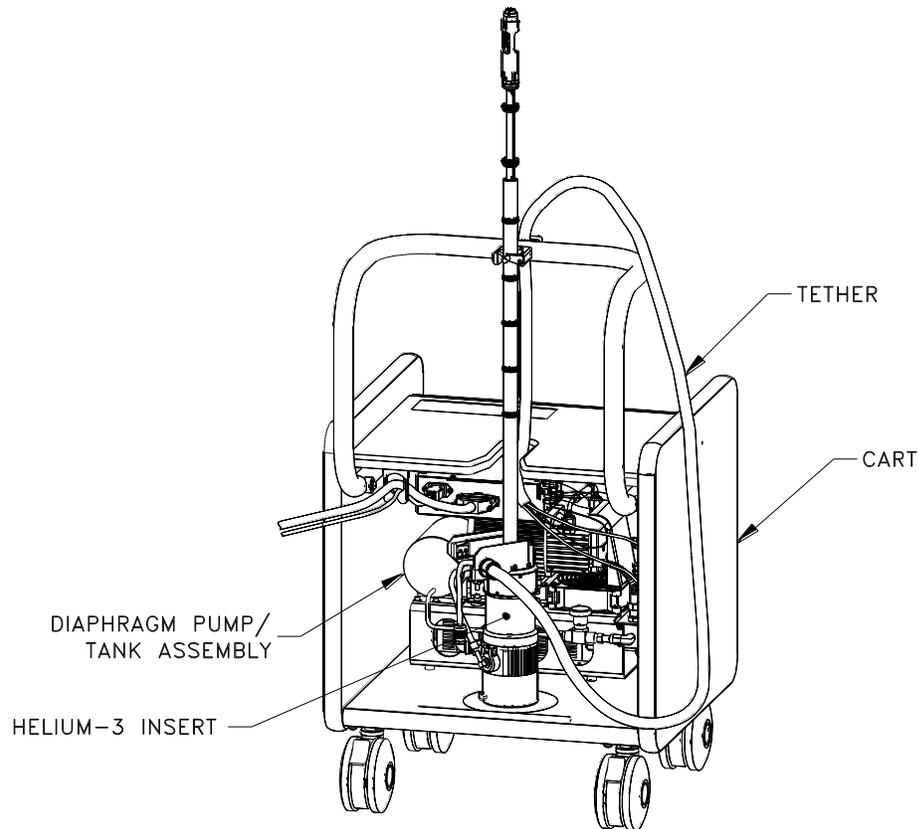


Figure A-2: Helium-3 Cart Assembly

6. Attach the tether, which is the black cable running between the diaphragm pump assembly and the 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.
7. 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 Helium-3 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.
8. Store the pump-out hose (part number 4092-630) inside the cart by hanging it from the cable hanger located on the right side next to where the handle is attached.
9. Place the step stool included with the Helium-3 option at the base of the PPMS dewar.

A.2.6 Connect the Cables

1. Connect the Helium-3 power cable (part number 3092-401) between the power connector on the diaphragm pump assembly and the power connector in the back of the PPMS electronics cabinet or to another power supply.
2. Connect the large connector on the black control cable (part number 3092-355) to the mating connector on the diaphragm pump connector. Connect the 25-pin connector to the splitter on the “P8–Auxiliary” connector at the back of the Model 6000. Connect the 9-pin serial cable connector to the COM1 port on the back of the computer.

3. Use the cable hanger located inside the Helium-3 cart on the left-hand side to hold both the control cable and the power cable.

A.2.7 Adjust the Helium-3 Insert for the Sample Chamber Orientation

Before the Helium-3 insert can be used, it must be adjusted so that it fits the specific PPMS sample chamber. The first adjustment is setting the orientation of the electrical connector at the bottom of the probe to match the orientation of the mating connector in the sample chamber.

Complete the following steps to verify or set the connector orientation:

1. Verify that the sample chamber temperature is 300 K and that the chamber is venting continuously.
2. Connect the user bridge cable with the gray Lemo connector between the “P1–User Bridge” port on the back panel of the Model 6000 and the gray color-coded connector at the head of the PPMS dewar. The user bridge cable will be used to read the Helium-3 thermometer.
3. Start up the PPMS MultiVu software.
4. Select **Instrument** ► **Bridge Channels** in PPMS MultiVu. Turn on bridge channel 3. Keep the **Bridge Channels** dialog box open.

CAUTION!

Avoid stressing the shaft of the refrigerator probe in any way while you are installing the Helium-3 insert into or removing it from the sample chamber. The material out of which the shaft is constructed is thin and fragile and can easily bend or dent.

5. Test the orientation of the electrical connector at the bottom of the refrigerator probe as follows:
(a) insert the refrigerator probe into the sample chamber and (b) monitor the resistance on user bridge channel 3. If the resistance on channel 3 is a consistent value between 30 and 60 ohms, the connector does not require adjustment. You may skip the following step. If the resistance is not a valid value, the connector must be adjusted by continuing with the next step.
6. Adjust the electrical connector at the bottom of the refrigerator probe if necessary. Do the following:
 - a. Remove the Helium-3 insert from the sample chamber.
 - b. Use a flashlight to look into the sample chamber. Note the orientation of the keyway at the bottom of the chamber. The keyway should face directly toward the front of the PPMS dewar. The front of the dewar faces away from all electrical connectors and hoses on the probe head. If the keyway does not face the front of the dewar, you must adjust the electrical connector on the refrigerator probe so that the connectors on the probe and in the sample chamber line up.
 - c. Use the smallest hex wrench included with the Helium-3 kit to loosen the three set screws inserted in the holes closest to the electrical connector on the refrigerator probe. Gently twist the electrical connector the correct amount in the correct direction so that it will line up with the connector in the sample chamber. Then retighten the set screws.



WARNING!

Do not twist the electrical connector on the refrigerator probe more than a few degrees. Twisting the connector more than a few degrees will permanently damage the wiring assembly. Using any appreciable force to twist the connector should be unnecessary.

- d. Test the orientation of the electrical connector by inserting the refrigerator probe into the sample chamber and monitoring the resistance on user bridge channel 3. Refer to step 5.

A.2.8 Cryoclean the Helium-3 Gas

The ^3He gas often becomes somewhat contaminated during shipment of the Helium-3 system. Therefore, you should cryoclean the gas before attempting to use the system. Follow the procedure “Clean He3 with LN2 Trap” (Section 6.3.1).

A.2.9 Test the Helium-3 System

1. Put a film of Apiezon N Grease on the contact fingers on the sample housing tube.
2. Insert the refrigerator probe into the sample chamber. You should feel resistance to the insertion of the probe in the bottom 4–5 cm. It should be enough to just support the weight of the probe.
3. Once the O-ring is firmly seated, purge the sample chamber.
4. Start the Helium-3 software from the Windows **Start** menu. The Helium-3 software enters a standby state.
5. Use the Model 6000 front panel or PPMS MultiVu to set a temperature of 1 K. Setting this first temperature causes the Helium-3 program to perform a self-check before proceeding down to 1 K. Depending on the probe, it takes from 2 to 3 hours to reach this temperature.
Once the system reaches 1 K, it should remain stable at that temperature and the Helium-3 software should indicate that it is in the He3 circulating state.
6. Verify that the system holds 1 K adequately. Then enter a set point of 0.31 to determine the base temperature of the instrument. It usually takes less than 30 minutes to reach the base temperature below 0.4 K. The status display in the Helium-3 option control console indicates it is in the “circulating” mode until the pot is filled, and then it enters the “one-shot” mode where it will reach the ultimate base temperature.

Note: If the system does not cool to below about 1.7 K, the system may be plugged and should be cryocleaned to remove contamination. Cryocleaning the system is a routine procedure described in Section 5.3.1. You may start the procedure with the system already cold and the insert installed.

Installation Instructions

B.1 Introduction

This appendix contains the following information:

- Section B.2 explains how to install the Helium-3 option in Physical Property Measurement System DynaCool platform.

B.2 Installing the Helium-3 Option

You must have the following tools to install the Helium-3 option:

- Computer running a current version of the DynaCool MultiVu software. Current version can be found at www.qdusa.com
- Cryopump high-Vacuum system
- 9/64-inch hex wrench
- 3/16-inch nut driver
- 9/32-inch nut driver
- Philips and standard screwdrivers

B.3 Install the Software

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

To install the software through CD, follow the printed instructions that are provided with the software CD. If no separate printed instructions are included, perform the following steps:

1. Install DynaCool MultiVu. Do the following: (a) insert DynaCool MultiVu software CD into the PC, (b) select the D: drive, (c) run `setup.exe` from the installation CD, and then (d) follow the on-screen instructions.
2. Install the Helium-3 option software. Do the following: (a) insert Helium-3 Installation CD into the PC, (b) select the D: drive, (c) run `setup.exe` from the installation CD, and then (d) follow the on-screen instructions. Insert the configuration disk when you are prompted to do so.
3. The Helium-3 software consists of the installation CD, which contain the program and utilities, configuration files and Helium-3 temperature calibration in SRAM files for the Helium-3 insert.
4. Install the Heat Capacity software, version 3.8.6 or later, if you will be using the Heat Capacity option (Model P650). Refer to the procedures in step 2.
5. Install the Electrical Transport Option software, version 1.1.35 or later, if you will be using the Electrical Transport Option (Model P605). Refer to the installation procedure in the ETO User's Manual.

B.4 Install CM-P Helium Control Module

The control electronics for the Helium-3 Option are contained in the Model CM-P Helium-3 Control Module. Refer to Section 2.4 for a drawing and a detailed description of this module. This Control Module normally resides in one of the module bays on the DynaCool CAN Module Bay on the side of the cryostat and may already have been installed.

If the Helium-3 Control Module is not already installed, then do this now:

1. Turn off all the Dynacool Services

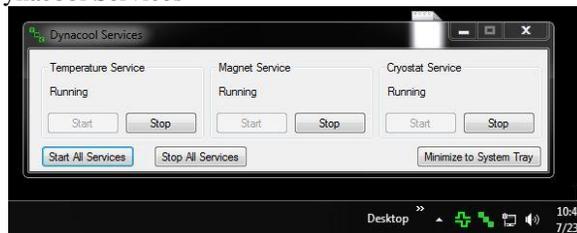


Figure B-1: Dynacool Services

2. Turn off the power to the DynaCool Module Bay and verify that there are no lit indicator lights on any of the installed modules.
3. Remove the cover plate from one of the unused module slots and insert the Model CM-E Heat Capacity Module into the empty slots.



WARNING!

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

4. Once the module is inserted and the mounting thumbscrews screwed in, turn on the power to the Module Bay.
5. Once the module self-test is complete, the PWR LED will be lit solid green and the COP LED will be solid or blinking green. This indicates normal hardware startup.
6. The front panel of the module has two LEDs in the top left, as shown in Figure 2-7. The PWR LED indicates the power-on status of the module. The COP (CANopen Protocol) LED indicates the status of the CAN network controller. Table B-1 outlines the LED states and provides solutions in the event of a problem.

Important: The error information in Table B-1 refers to situations that persist for longer than about 15 seconds. Typically, when the module is powered on, the LEDs may briefly flash red before they turn green. This is a normal part of the startup or reset sequence.

Table B-1: LED guide for Model CM-P He-3 Control Module

LED	COLOR	STATUS	MEANING AND/OR SOLUTION
PWR	Green	On	The processor is running with no errors (normal).
	Red	Flashing	Errors were encountered during the self-test. The flashing sequence can be used to determine the cause of the failure.
COP	Green	On	CAN status is operational (normal).
		Flashing	CAN status is pre-operational. Verify that cable is connected to PC.
	Red	On or flashing	Error on the CAN bus. Contact Quantum Design for assistance.

If you are unable to achieve operation with both LEDs green, please contact Quantum Design for assistance.

7. Turn on all the Dynacool Services which we turned off earlier.

B.5 Place the Scroll Pump Assembly in the Cart

CAUTION!

It is strongly recommended that at least two people handle the pump and probe assembly while completing the following procedure. The Helium-3 refrigerator probe is very fragile. The thin wall of the probe shaft can be easily dented or bent, and the turbo pump mounted on the shaft is very sensitive to mechanical shocks. The backing pump assembly contains many gas-tight seals that could be compromised if the backing pump is dropped or handled roughly.

1. Remove the backing pump assembly and the attached Helium-3 insert from the packing crate.
2. Verify that the power selector on the backing pump assembly is set to the proper line voltage (for example, 110 V or 220 V). To set the voltage, remove the fuse holder and change the selector. The fuse holder is part of the power connect located on the tank end of the controller.
3. Lay the Helium-3 insert on the floor or have someone hold it.
4. Carefully lift up the backing pump assembly. Reattach any electrical connectors that were unplugged for shipping.
5. Carefully place the backing pump assembly at the back of the open cabinet area in the Helium-3 cart. Position the tank so that its four rubber feet fit into the four holes in the floor of the cabinet. Refer to Figure B-2. Work with extreme care to avoid torquing, crimping, or loosening any of the attached hoses.



Figure B-2: Helium-3 Cart Assembly

6. Attach the tether, which is the black cable running between the backing pump assembly and the 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.
7. 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 Helium-3 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.
8. Store the pump-out hose (part number 4092-630) inside the cart by hanging it from the cable hanger located on the right side next to where the handle is attached.
9. Place the step stool included with the Helium-3 option at the base of the PPMS dewar.

B.6 Connect the Cables

1. Connect the Helium-3 power cable (part number 3092-401) between the power connector on the backing pump assembly and the power connector in the back of the PPMS electronics cabinet or to another power supply.
2. Connect the large connector on the black control cable (part number 3092-355) to the mating connector on the backing pump connector. Connect the 25-pin connector to the Helium-3 CAN Module and the Gas Handling System. Refer to Tables B-2 and B-3 on the following pages.
3. Use the cable hanger located inside the Helium-3 cart on the left-hand side to hold both the control cable and the power cable.

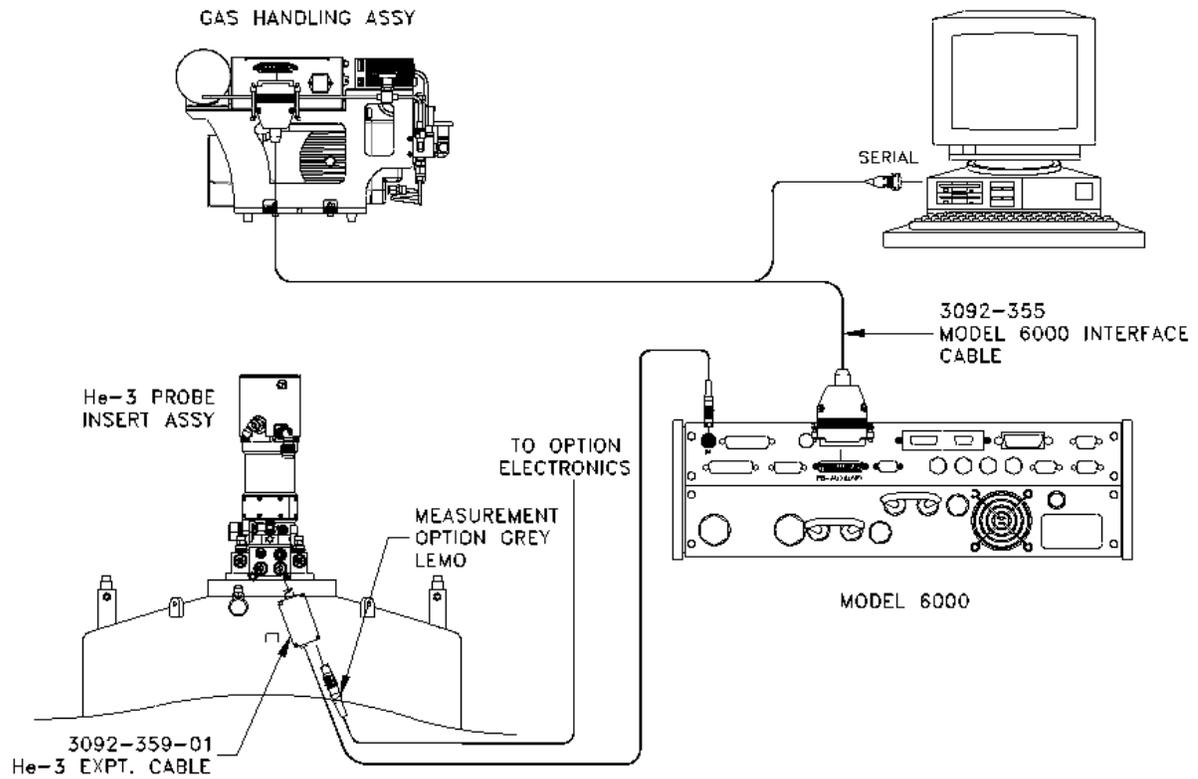


Figure B-3A: System Cabling/Block Diagram

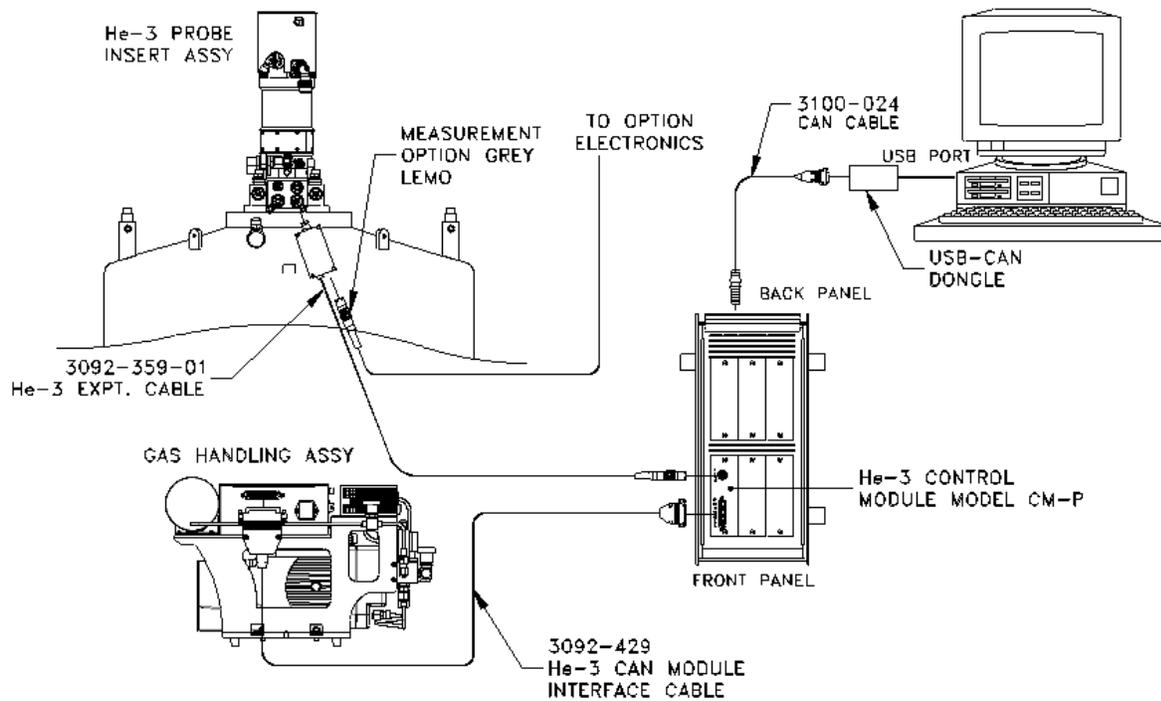


Figure B-4B: System Cabling/Block Diagram

Table B-2: Interconnection Table for Helium-3 CAN Module Interface Cable (Part Number 3092-429)

25-PIN/COAX D-SHELL CONNECTOR AT P7 ON BACKING/SCROLL PUMP ASSEMBLY	25-PIN D-SHELL CONNECTOR AT P2 ON HE3 CAN MODULE	DESCRIPTION
2	7	Serial port RXD
3	20	Serial port TXD
5	19	Serial port GND
10	15	Pressure gauge supply (+15 VDC)
11	2	Pressure gauge read back voltage (VDC+)
12	18	Bypass solenoid driver (+24 VDC)
13	5	Supply solenoid driver (+24 VDC)
22	No Connection	Helium-3 control wiring shield
23	4	Pressure gauge return,
23	21	Probe-sealed switch com
24	8	Probe-sealed switch signal
25	17	Solenoid drivers return
A1-center	No connection	Helium-3 heater
A1-shield	No connection	Helium-3 heater coax shield

Table B-3: Pin Mapping for Helium-3 Thermometer Wiring with the Experiment Cable

HELIUM-3 PROBE CONNECTOR	GRAY LEMO CONNECTOR ON PROBE HEAD	GRAY LEMO CONNECTORS ON ETO SAMPLE, HE3 CABLE (3101-359)			ETO SAMPLE CABLE (3101-456)		FUNCTION
		14 PIN PLUG	14 PIN SOCKET	4 PIN PLUG	14 PIN SOCKET	8 PIN FISHER PLUG	
3	3	3	3	-	3	1	User/resist ch1 I+
4	4	4	4	-	4	8	User/resist ch1 I-
5	5	5	5	-	5	4	User/resist ch1 V+
6	6	6	6	-	6	5	User/resist ch1 V-
7	7	7	7	-	7	6	User/resist ch2 I+
8	8	8	8	-	8	2	User/resist ch2 I-
9	9	9	9	-	9	7	User/resist ch2 V+
10	10	10	10	-	10	3	User/resist ch2 V-
11	11	11	N/C	1	N/C	-	Helium-3 therm I+
12	12	12	N/C	2	N/C	-	Helium-3 therm I-
13	13	13	N/C	3	N/C	-	Helium-3 therm V+
14	14	14	N/C	4	N/C	-	Helium-3 therm V-

B.7 Adjust the Helium-3 Insert for the Sample Chamber Orientation

Before the Helium-3 insert can be used, it must be adjusted so that it fits the specific PPMS sample chamber. The first adjustment is setting the orientation of the electrical connector at the bottom of the probe to match the orientation of the mating connector in the sample chamber.

Complete the following steps to verify or set the connector orientation:

1. Verify that the sample chamber temperature is 300 K and that the chamber is venting continuously.
2. Connect the user bridge cable with the gray Lemo connector between the “P1–User Bridge” port on the CAN module and User Experiment splitter box. The user bridge cable will be used to read the Helium-3 thermometer.
3. Start up the PPMS MultiVu software.
4. Select **Instrument** ► **Bridge Channels** in PPMS MultiVu. Turn on bridge channel 3. Keep the **Bridge Channels** dialog box open.

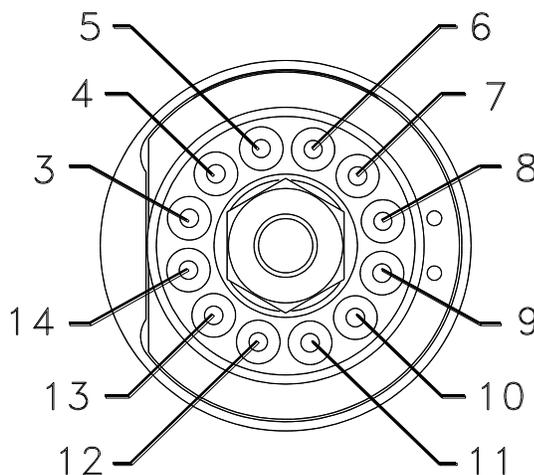
CAUTION!

Avoid stressing the shaft of the refrigerator probe in any way while you are installing the Helium-3 insert into or removing it from the sample chamber. The material out of which the shaft is constructed is thin and fragile and can easily bend or dent.

5. Test the orientation of the electrical connector at the bottom of the refrigerator probe as follows: (a) insert the refrigerator probe into the sample chamber and (b) monitor the resistance on user bridge channel 3. If the resistance on channel 3 is a consistent value between 30 and 60 ohms, the connector does not require adjustment. You may skip the following step. If the resistance is not a valid value, the connector must be adjusted by continuing with the next step.
6. Adjust the electrical connector at the bottom of the refrigerator probe if necessary. Do the following:
 - a. Remove the Helium-3 insert from the sample chamber.
 - b. Use a flashlight to look into the sample chamber. Note the orientation of the keyway at the bottom of the chamber. The keyway should face directly toward the front of the PPMS dewar. The front of the dewar faces away from all electrical connectors and hoses on the probe head. If the keyway does not face the front of the dewar, you must adjust the electrical connector on the refrigerator probe so that the connectors on the probe and in the sample chamber line up.
 - c. Use the smallest hex wrench included with the Helium-3 kit to loosen the three set screws inserted in the holes closest to the electrical connector on the refrigerator probe. Gently twist the electrical connector the correct amount in the correct direction so that it will line up with the connector in the sample chamber. Then retighten the set screws.

	WARNING!
<p>Do not twist the electrical connector on the refrigerator probe more than a few degrees. Twisting the connector more than a few degrees will permanently damage the wiring assembly. Using any appreciable force to twist the connector should be unnecessary.</p>	

- d. Test the orientation of the electrical connector by inserting the refrigerator probe into the sample chamber and monitoring the resistance on user bridge channel 3. Refer to step 5.



BOTTOM VIEW, PROBE CONNECTOR

Figure B-5: Bottom View of Probe Connector

B.8 Cryoclean the Helium-3 Gas

The ³He gas often becomes somewhat contaminated during shipment of the Helium-3 system. Therefore, you should cryoclean the gas before attempting to use the system. Follow the procedure “Clean He3 with LN2 Cold Trap” (Section 6.3.1).

B.9 Test the Helium-3 System

1. Put a film of Apiezon N Grease on the contact fingers on the sample housing tube.
2. Insert the refrigerator probe into the sample chamber. You should feel resistance to the insertion of the probe in the bottom 4–5 cm. It should be enough to just support the weight of the probe.
3. Once the O-ring is firmly seated, purge the sample chamber.
4. Start the Helium-3 software from the Windows **Start** menu. The Helium-3 software enters a standby state.
5. Use PPMS MultiVu to set a temperature of 1 K. Setting this first temperature causes the Helium-3 program to perform a self-check before proceeding down to 1 K. Depending on the probe, it takes from 2 to 3 hours to reach this temperature.

Once the system reaches 1 K, it should remain stable at that temperature and the Helium-3 software should indicate that it is in the He3 circulating state.

6. Verify that the system holds 1 K adequately. Then enter a set point of 0.31 K to determine the base temperature of the instrument. It usually takes less than 30 minutes to reach the base temperature below 0.4 K. The status display in the Helium-3 option control console indicates it is in the “circulating” mode until the pot is filled, and then it enters the “one-shot” mode where it will reach the ultimate base temperature.

Note: If the system does not cool to below about 1.7 K, the system may be plugged and should be cryocleaned to remove contamination. Cryocleaning the system is a routine procedure described in Section 6.3.1. You may start the procedure with the system already cold and the insert installed.

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