

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



Physical Property Measurement System[®]

Heat Capacity Option User's Manual

Part Number 1085-150, Rev. M7

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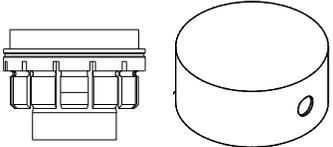
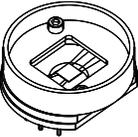
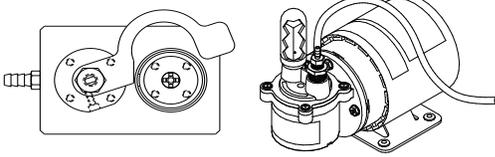
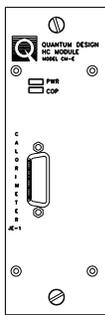
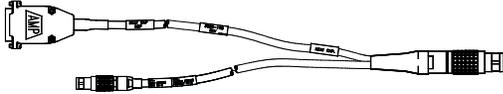
U.S. Patents

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

Foreign Patents

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

Components of the Heat Capacity Option

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

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Contents and Conventions

P.1 Introduction

This preface contains the following information:

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

P.2 Scope of the Manual

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

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

P.3 Contents of the Manual

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

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

P.4 Conventions in the Manual

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

CAUTION!

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



WARNING!

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



WARNING!

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

Overview of the Heat Capacity System

1.1 Introduction

This chapter contains the following information:

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

1.2 What the System Measures

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

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

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

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

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

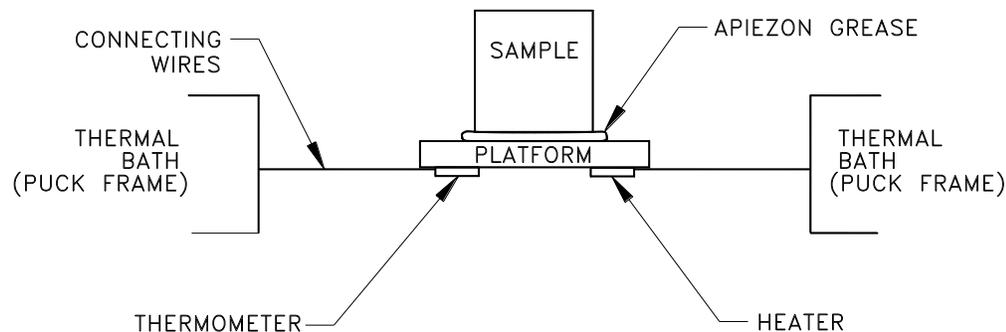


Figure 1-1. Thermal Connections to Sample and Sample Platform in PPMS Heat Capacity Option

1.3 Purpose of Measuring Heat Capacity

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

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

1.4 Scope of the Heat Capacity Option

1.4.1 Sample Size and Thermal Characteristics

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

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

1.4.2 Temperature Range

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

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

1.4.3 Measurement Technique

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

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

1.4.4 Pressure in Sample Chamber

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

1.5 Special Features of the Heat Capacity Option

1.5.1 Acquisition Hardware

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

1.5.2 Calorimeter Puck and Sample Mounting

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

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

1.5.3 Puck Calibration

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

1.5.4 Automation Environment

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

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

Installation and Getting Started

2.1 Introduction

This chapter contains the following information:

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

2.2 System Requirements

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

2.3 Installing the Hardware and Software on a PPMS

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

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

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

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

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

2.3.1 Install the Software

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

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

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

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

2.3.2 Install the Heat Capacity Control Electronics and Cable

2.3.2.1 INSTALL THE MODULE 1000 TOWER

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

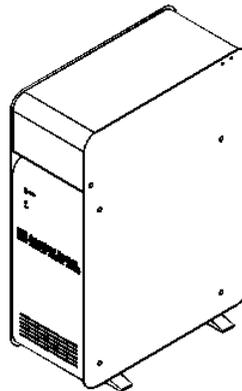


Figure 2-1 External View of the Model 1000 Modular Control System (4100-001)

2.3.2.2 INSTALL THE CM-E HEAT CAPACITY CONTROLLER MODULE

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

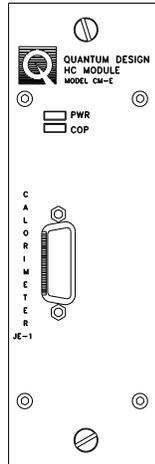


Figure 2-2 Heat Capacity CAN Module

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

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



WARNING!

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

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

2.3.2.3 CONNECT THE CONTROLLER CABLE

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

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

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

2.3.3 Set Up the Sample-Mounting Station



WARNING!

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

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

2.4 Performing a First Addenda Measurement

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

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

Hardware

3.1 Introduction

This chapter contains the following information:

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

3.2 Heat Capacity Hardware

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

3.2.1 Calorimeter Puck

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

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

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

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

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

3.2.1.1 PUCK FRAME

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

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

3.2.1.2 THERMAL RADIATION SHIELD

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

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

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

3.2.1.3 SERIAL NUMBER

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

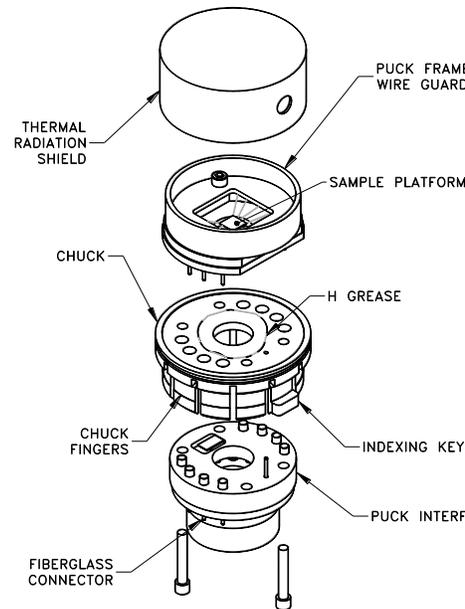


Figure 3-1. Exploded View of Calorimeter

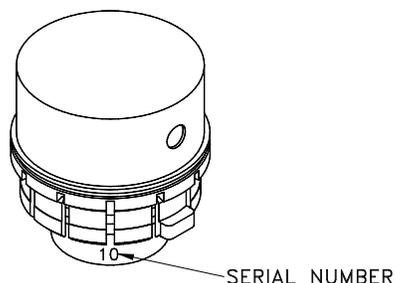


Figure 3-2. Location of Serial Number on Calorimeter Puck

3.2.2 Sample-Mounting Station

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

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

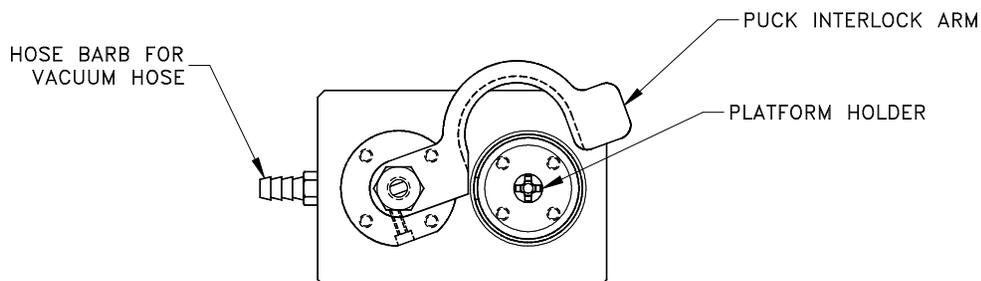


Figure 3-3. Top View of Sample-Mounting Station

3.2.2.1 VACUUM PUMP

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



WARNING!

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

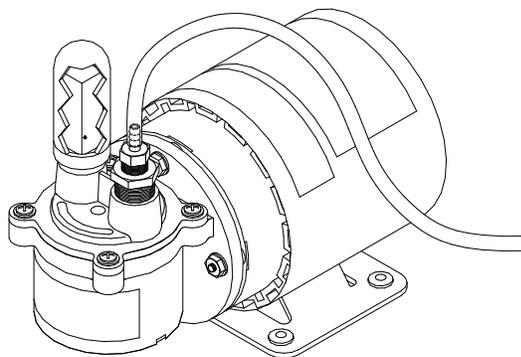


Figure 3-4. Vacuum Pump for Sample-Mounting Station

3.2.3 Model CM-E Heat Capacity Control Module

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

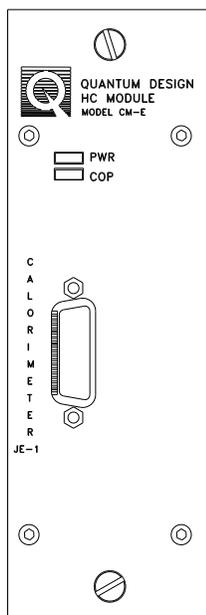


Figure 3.5 Heat Capacity CAN Module

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

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

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

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

3.2.4 Controller Cable Assembly

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

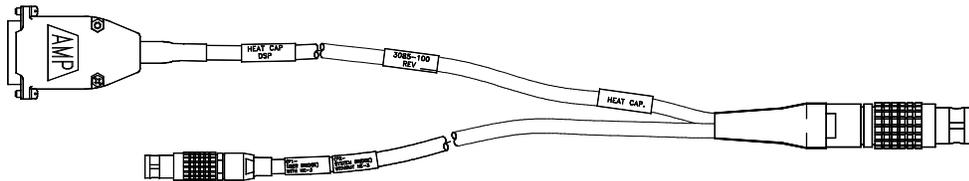
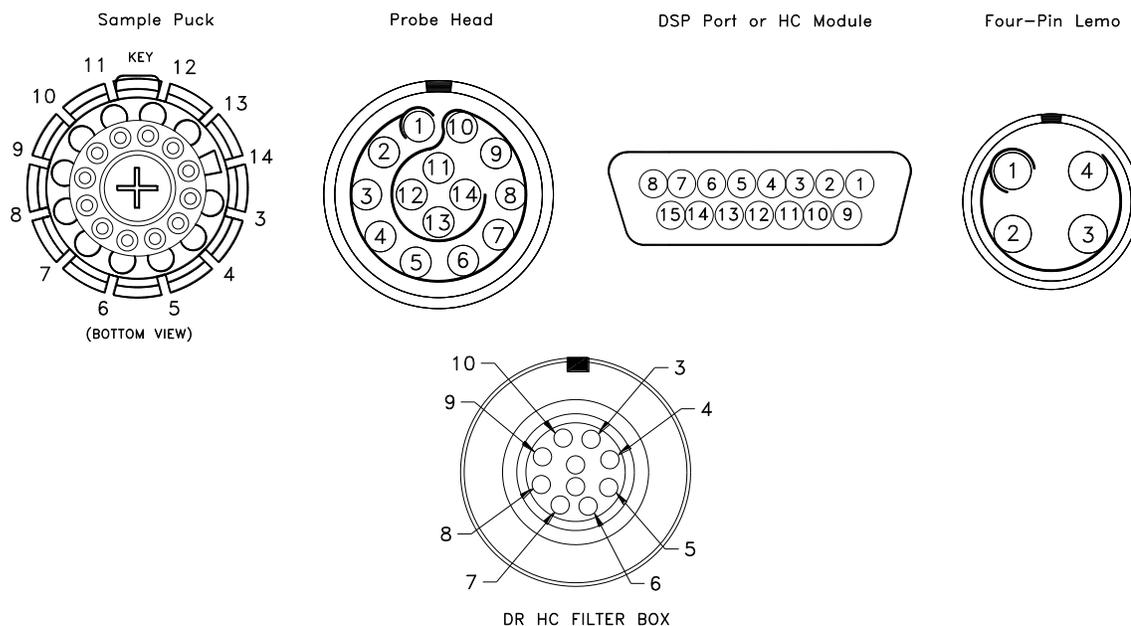


Figure 3-6 Controller Cable

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

Table 3-1. Sample Connections for Pin Numbers



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

3.3 High-Vacuum Hardware

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

3.3.1 Contact Baffle for Standard Heat Capacity

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

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

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

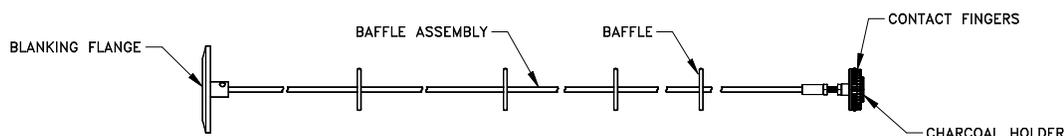
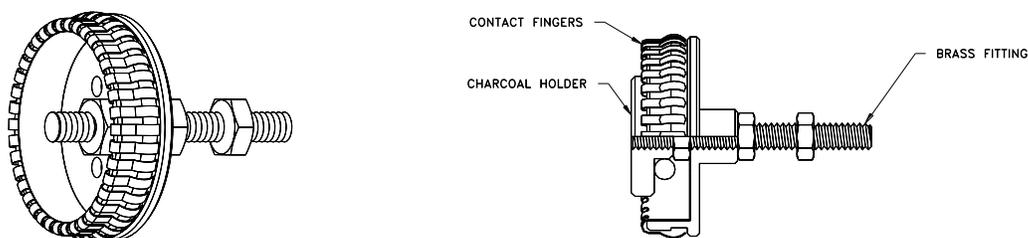


Figure 3-7. Baffle Assembly with Contact Baffle



A. Charcoal Holder Removed for Puck Calibration

B. Charcoal Holder Installed for Normal Operation

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

Software

4.1 Introduction

This chapter contains the following information:

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

4.2 Overview of System Software

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

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

Table 4-1. Software Files for Heat Capacity Option

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

4.3 Analysis Models

4.3.1 Thermal Models

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

4.3.1.1 SIMPLE MODEL

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

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

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

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

4.3.1.2 TWO-TAU MODEL

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

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

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

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

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

4.3.2 Data Fitting

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

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

4.3.3 Derived Quantities from Model Parameters

4.3.3.1 SIMPLE MODEL: ADDENDA MEASUREMENT

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

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

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

4.3.3.2 SIMPLE MODEL: SAMPLE MEASUREMENT

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

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

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

4.3.3.3 TWO-TAU MODEL: SAMPLE MEASUREMENT

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

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

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

The expressions for α and β are given by

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

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

4.3.3.4 EQUIVALENT DEBYE TEMPERATURE

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

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

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

$$C_v = 3rNk \int_0^{\infty} \left(\frac{hv}{2kT} \right)^2 \csc h^2 \left(\frac{hv}{2kT} \right) g(v) dv,$$
(4.4)

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

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

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

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

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

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

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

which is the familiar T^3 law.

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

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

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

4.3.3.5 MEASUREMENT UNITS

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

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

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

4.3.3.6 ESTIMATED HEAT CAPACITY ERROR

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

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

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

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

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

4.3.4 Slope Analysis of Relaxation Curves

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

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

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

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

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

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

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

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

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

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

4.3.4.2 DUAL-SLOPE ANALYSIS (LARGE TEMPERATURE RISES)

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

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

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

4.3.4.3 OPTIMIZING MEASUREMENTS FOR SLOPE-ANALYSIS

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

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

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

4.4 Heat Capacity Control Center

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

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

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

4.4.1 Installation Wizards Tab

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

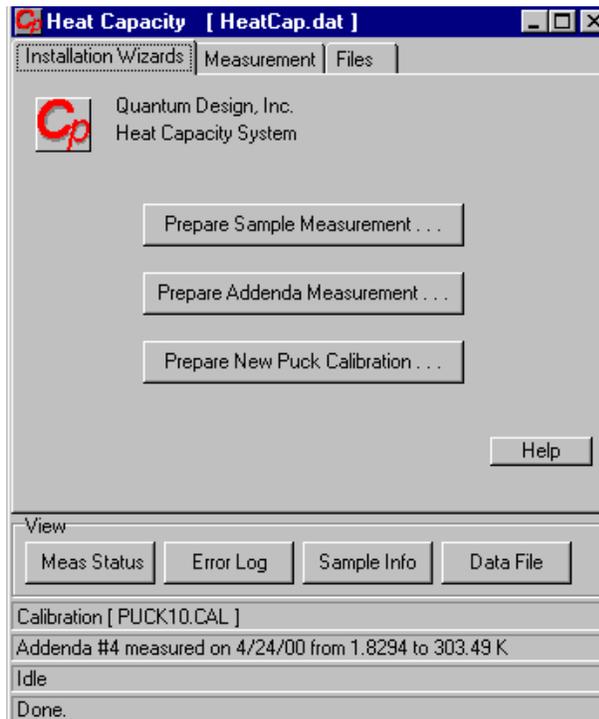


Figure 4-1. **Installation Wizards** Tab in Heat Capacity Control Center

4.4.2 Measurement Tab

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

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

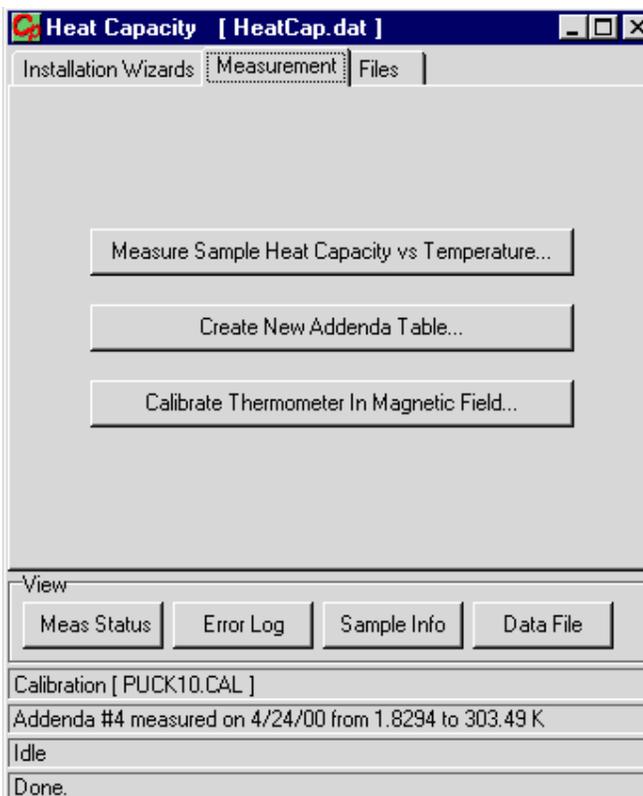


Figure 4-2. **Measurement** Tab in Heat Capacity Control Center

4.4.3 Files Tab

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

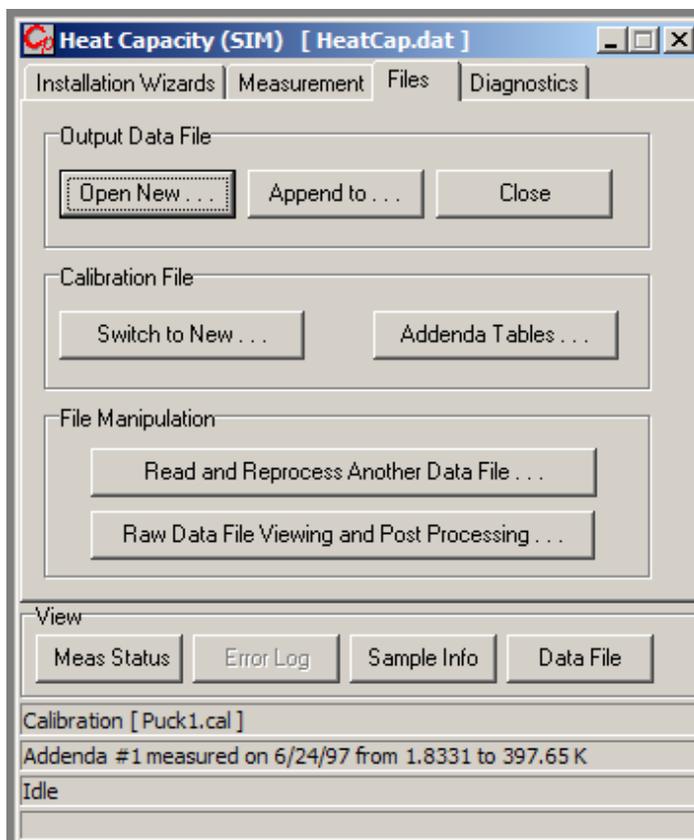


Figure 4-3. **Files** Tab in Heat Capacity Control Center

4.5 Measurement Status Viewer

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

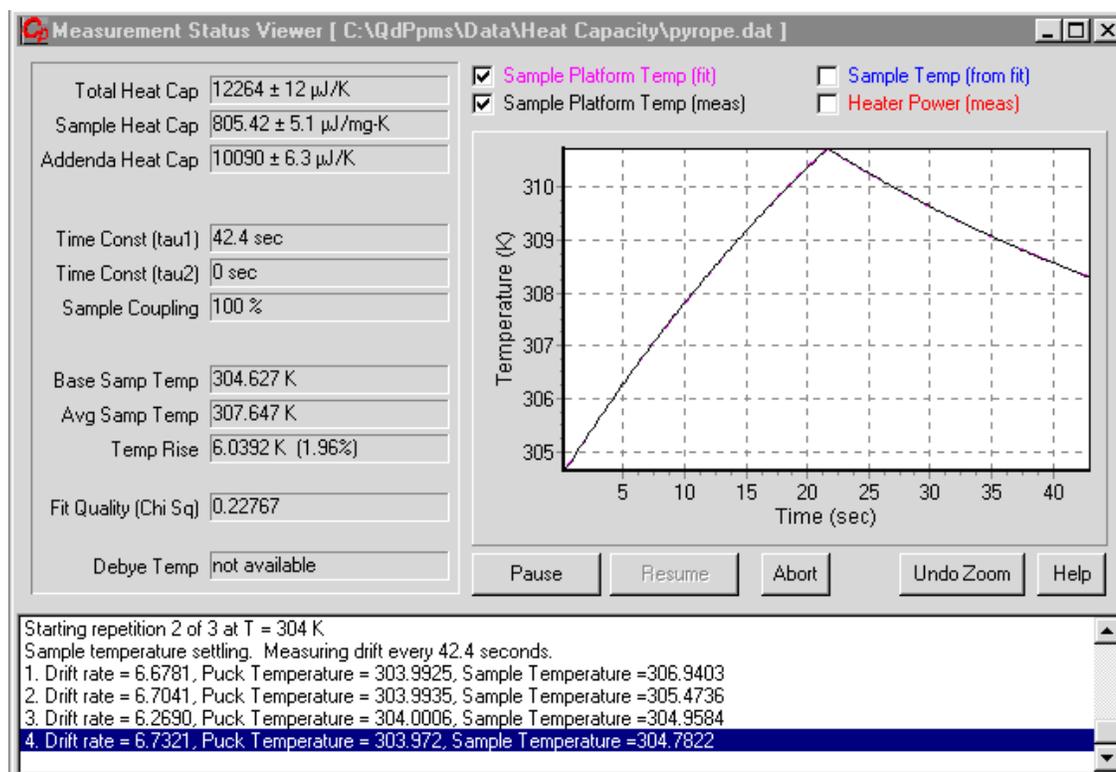


Figure 4-4. Measurement Status Viewer. The graph on the right side of the Viewer displays a plot of fit and measured temperature versus time.

4.5.1 Measurement Fields

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

Table 4-6. Measurement Fields in Measurement Status Viewer

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

(table continues)

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

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

4.5.2 Graph

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

Table 4-7. Temperature Data Viewing Options

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

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

Panning through the graph.

To pan through the graph, hold down the right mouse button and drag across the graph. You may drag from left to right or from right to left. You select the **Undo Zoom** button in the Measurement Status Viewer to return the graph to its original size.

4.5.3 Message List Box

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

4.6 Raw Data Browser and Slope Analysis

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

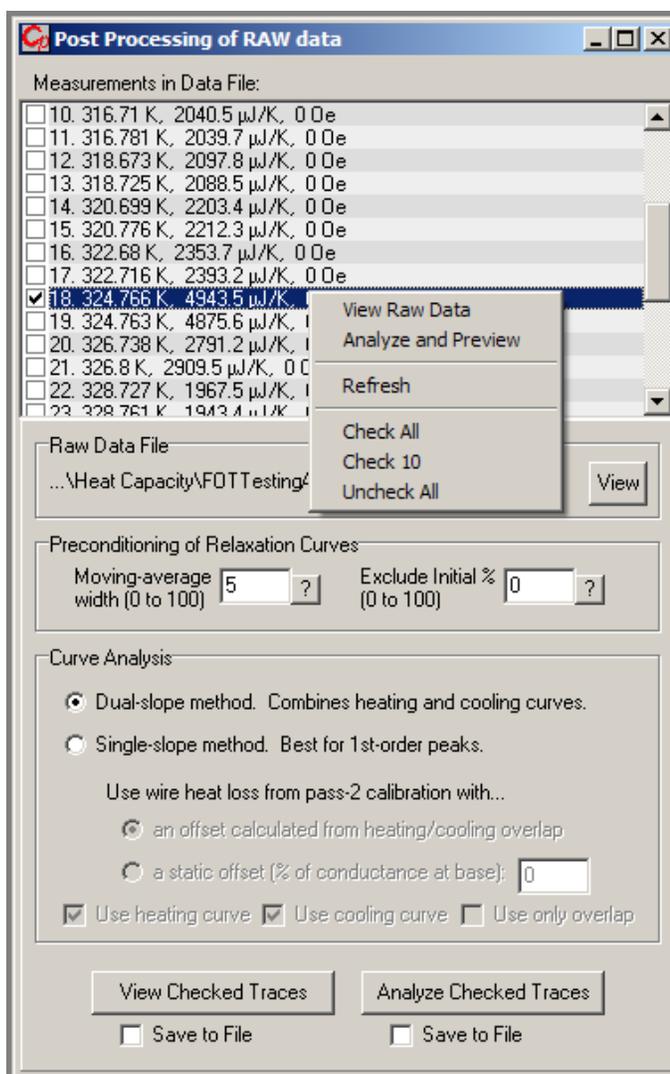


Figure 4-5. Post Processing of Raw Data dialog.

4.6.1 Browsing the Raw Data

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

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

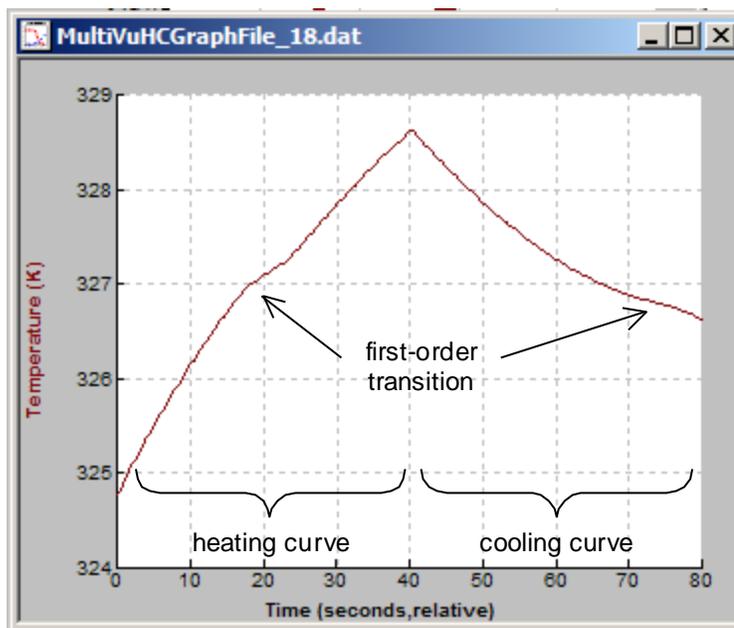


Figure 4-6. Example of raw temperature response data distorted near a first-order transition.

4.6.2 Slope Analysis

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

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

4.6.2.1 EXAMPLE: SINGLE-SLOPE ANALYSIS

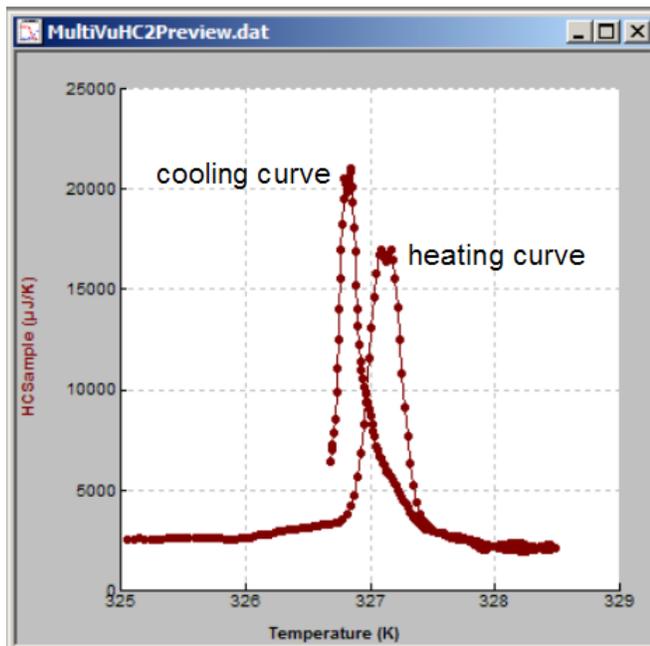


Figure 4-7. Example data from Fig. 4-6 analyzed using the single-slope analysis technique with a Moving Average Width of 10%

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

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

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

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

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

4.6.2.2 EXAMPLE: DUAL-SLOPE ANALYSIS

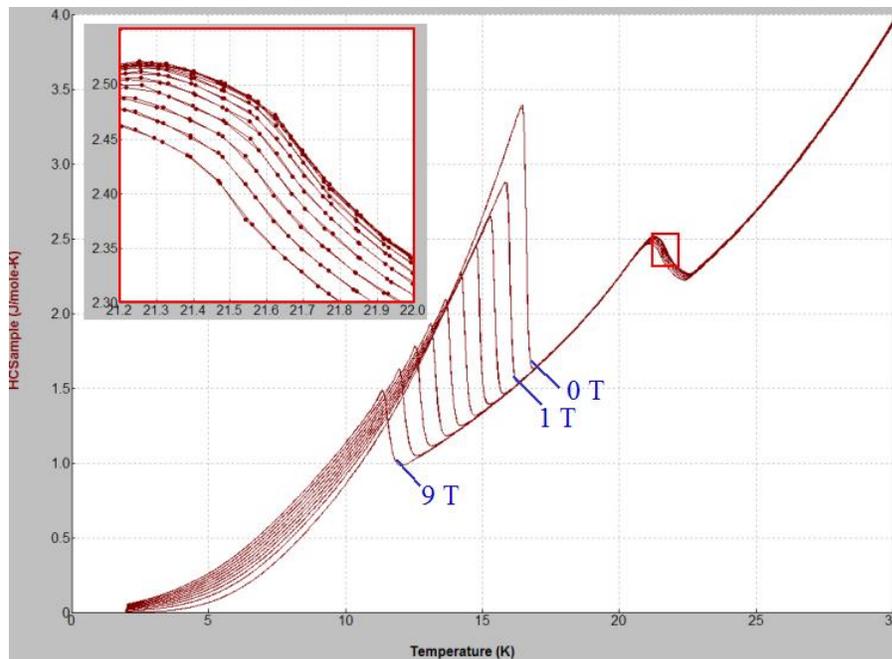


Figure 4-8. Heat Capacity from dual-slope method of a V_3Si superconductor in different magnetic fields. Inset shows the repeatability and resolution that is possible with this method.

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

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

4.7 Writing Sequences

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

Table 4-8. Heat Capacity Measurement Sequence Commands

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

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

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

4.8 Heat Capacity Data Files

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

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

4.8.1 New Datafile Sequence Command

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

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

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

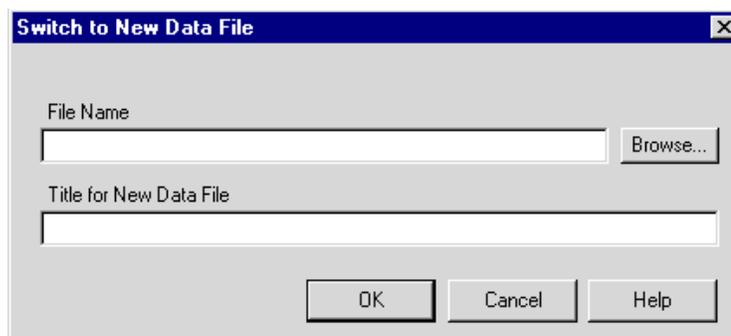


Figure 4-9. **Switch to New Data File** Dialog Box for **New Datafile** Sequence Command

4.8.2 Data File Header

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

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

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

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

- Data File Name . . .**: A text box containing "C:\QdPpms\Data\Heat Capacity\HeatCap.dat".
- Header Information . . .**: A text box containing "HeatCap.dat".
- Buttons**: "Open New File" and "Append to File" are located to the right of the text boxes.
- Table**: A table with three columns: "Name:", "Description:", and "Value:".

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

Figure 4-10. Data File Header

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

4.8.2.1 USER-DEFINED SAMPLE FIELDS

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

Creating a Sample Field.

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

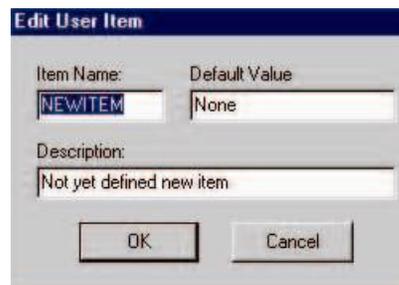


Figure 4-11. Edit User Item Dialog Box

Editing User-Defined Sample Fields.

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

Deleting User-Defined Sample Fields.

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

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

4.8.3 Format of Data Files

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

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

(table continues)

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

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

4.8.4 Format of .raw Files

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

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

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

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

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

4.9 Message and Error Log Files

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

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

Creating and Using Calibration Files

5.1 Introduction

This chapter contains the following information:

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

5.2 Overview of Puck Calibration

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

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

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

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

5.2.1 Puck Type

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

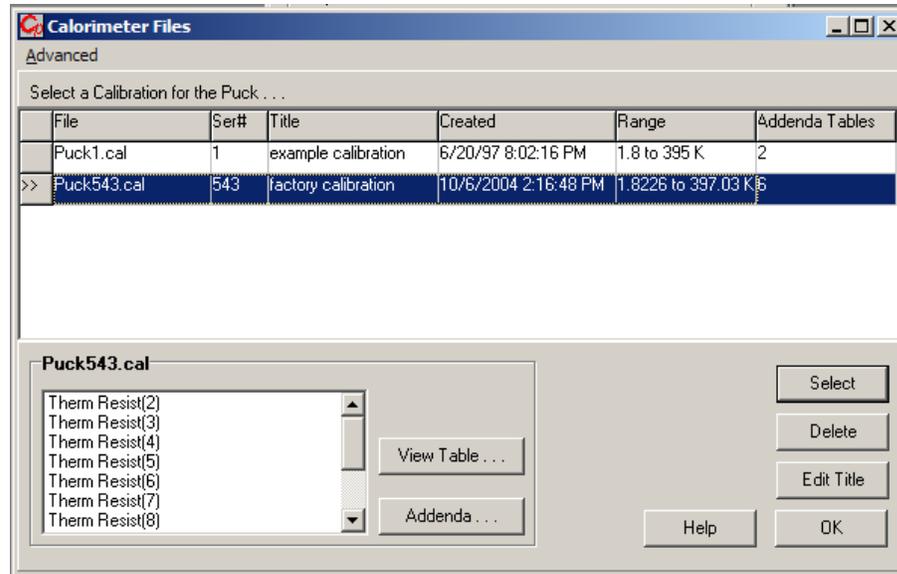
5.2.2 Calibration Files

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

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

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

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



File	Ser#	Title	Created	Range	Addenda Tables
He3Puck10.cal	10	full calibration	2/23/99 2:49:36 PM	no entries	0
>> PUCK10.CAL	10	factory calibration	6/20/97 6:26:47 PM	1.8 to 395 K	2

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

5.2.3 Active Calibration File

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

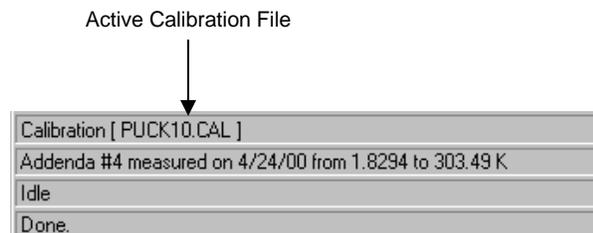


Figure 5-2. Active Calibration File Identified in **Calibration** Status Panel in Control Center

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

5.2.4 Calibration Tables

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

5.2.5 Addenda as Part of Calibration File

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

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

5.2.6 Use of Data Files

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

5.2.7 Magnetic Field Correction

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

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

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

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

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

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

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

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

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

5.3 Calibrating a Puck

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

5.3.1 Prepare the Puck

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

5.3.2 Insert the Puck and Remove the Charcoal Holder

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

CAUTION!

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

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

5.3.3 Test the Puck

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

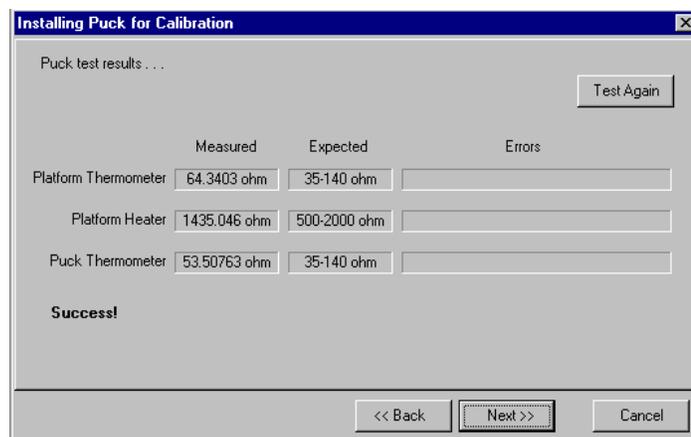


Figure 5-3. **Puck-test results** panel in puck installation wizard. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

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

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

5.3.4 Open a Data File

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

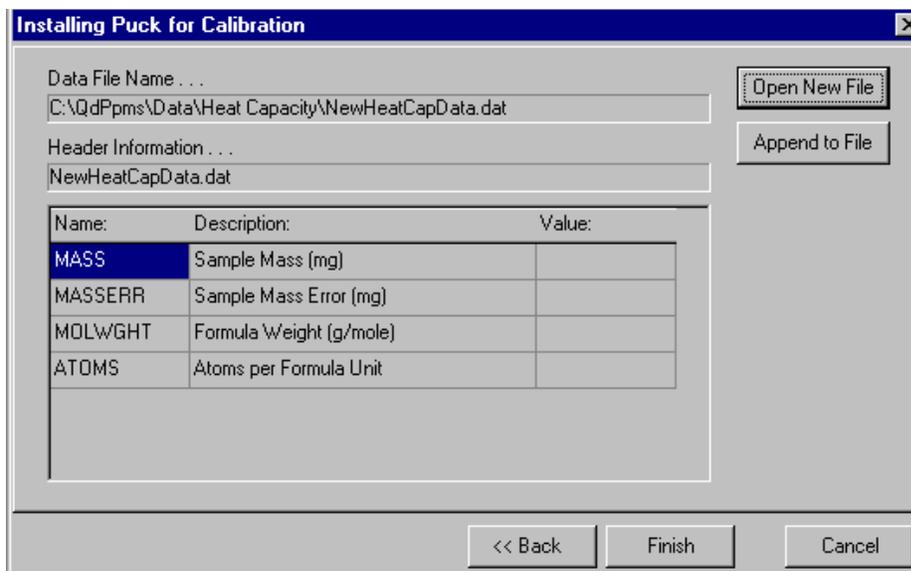


Figure 5-4. **Data File Name** panel in puck installation wizard. The name and location of the active data file are identified at the top of the panel. The information in the header of the active file is displayed below the **Header Information** box. If no data file is active, the **Data File Name**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

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

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

3. Enter a different title for the graph view of the file, if necessary. By default, the file name is also the title of the graph view.
4. Review the user-defined **Optional Information** that will be saved to the file header. Select **Configure List** to add, edit, or delete optional items. See Section 4.7.2.1 for more information.
When you create a new data file to store calibration data, you are not prompted to define any sample information that will be saved to the data file header. The application ignores sample information when it runs the calibration.
5. Select **OK** to close the **New Data File Information** dialog box. The **Data File Name** panel appears again. The **Header Information** now displays the header of the data file you have just opened.
6. Select **Finish** to close the **Data File Name** panel and open the **Puck Calibration (Pass 1)** dialog.

5.3.5 Run Pass 1 of the Calibration

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

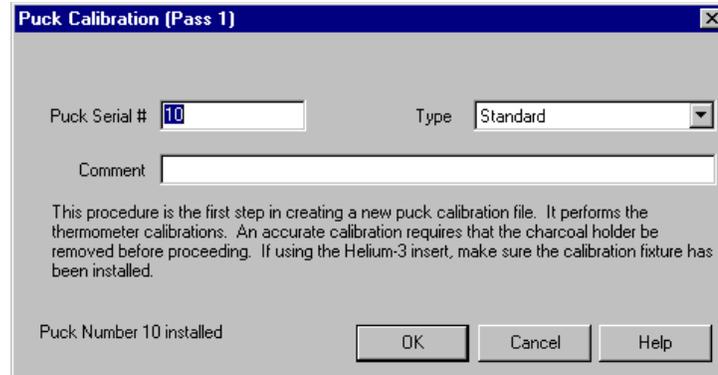


Figure 5-5. Puck Calibration (Pass 1) Dialog Box

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

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

5.3.5.1 PAUSING OR ABORTING A CALIBRATION

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

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

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

5.3.6 Run Pass 2 of the Calibration

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

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

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

5.4 Adding Magnetic Field Correction Tables to the Calibration

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

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

1. Install the Helium-3 calibration fixture as described in Section 8.5.1. Installing the calibration fixture ensures that the temperature of the sample platform will be as close as possible to the Helium-3 system thermometer.
2. Select the **Installation Wizards** tab in the Heat Capacity control center.
3. Select **Prepare Addenda Measurement** and follow the on-screen instructions to install the Helium-3 probe in the PPMS sample chamber, select the appropriate calibration file, test the puck, and specify an optional output file. It is assumed that the puck has already been calibrated in zero field so that a corresponding `.cal` file already exists.
4. Set the field for the PPMS to 90,000 Oe and wait until the field is achieved and the magnet is persistent.
5. Select the **Measurement** tab in the Heat Capacity control center.
6. Select **Calibrate Thermometer in Magnetic Field**.

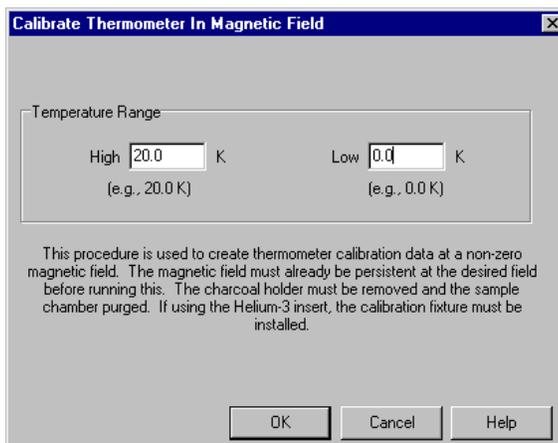


Figure 5-6. Calibrate Thermometer in Magnetic Field Dialog Box

7. Enter 20 K for the high temperature limit and 0.0 for the low temperature limit (see Figure 5-6). This will generate field correction data at this field from 20 K down to the lowest temperature in the calibration file.
8. Press **OK**. The procedure begins and will take several hours to complete.

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

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

5.5 Description of Calibration Tables

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

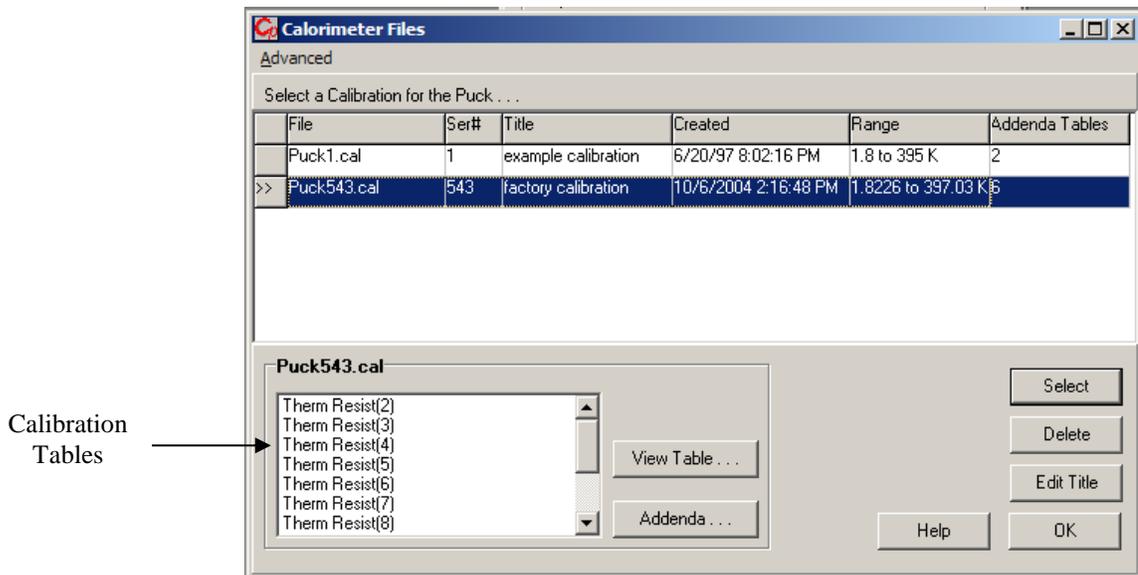


Figure 5-7. List of Calibration Tables in **Calorimeter Files** Dialog Box

5.5.1 Tables for Platform Thermometer Resistance

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

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

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

Figure 5-8. Table Displaying Platform Thermometer Resistance

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

5.5.2 Magnetic Field–Corrected Platform Thermometer Resistance

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

5.5.3 Table for Puck Thermometer Resistance

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

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

The screenshot shows a dialog box titled "Calibration Table Edit" with a menu bar containing "File". Below the menu bar is a table with two columns: "Temp" and "PuckRes". The table contains six rows of data. Below the table is a "Calculator" section with two input fields labeled "Temp" and "PuckRes", each with "(Ln pre-function)" below it. Between the fields are two buttons: "==" and "<==". At the bottom are "OK" and "Cancel" buttons.

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

Figure 5-9. Table Displaying Puck Thermometer Resistance

5.5.4 Tables for Platform Heater Resistance and Thermal Conductance

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

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

The screenshot shows a dialog box titled "Calibration Table Edit" with a menu bar containing "File". Below the menu bar is a table with two columns: "Temp" and "HtrRes". The table contains six rows of data. Below the table is a "Calculator" section with two input fields labeled "Temp" and "HtrRes", each with "(no pre-function)" below it. Between the fields are two buttons: "==" and "<==". At the bottom are "OK" and "Cancel" buttons.

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

Figure 5-10. Table Displaying Platform Heater Resistance

The screenshot shows a dialog box titled "Calibration Table Edit" with a menu bar containing "File". Below the menu bar is a table with two columns: "Temp" and "Conductance". The table contains six rows of data. Below the table is a "Calculator" section with two input fields labeled "Temp" and "Conductance", each with "(Ln pre-function)" below it. Between the fields are two buttons: "==" and "<==". At the bottom are "OK" and "Cancel" buttons.

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

Figure 5-11. Table Displaying Thermal Conductance

5.6 Description of Calibration Process

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

5.6.1 Pass 1

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

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

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

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

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

5.6.2 Pass 2

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

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

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

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

5.6.3 Magnetic Field Correction Process

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

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

5.7 Managing Calibration Files

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

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

5.7.1 Selecting a Calibration File

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

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

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

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

5.7.2 Editing the Title of a Calibration File

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

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

1. Open the **Calorimeter Files** dialog box (Figure 5-1 or 5-7) by doing one of the following: (a) double-click on the **Calibration** status panel in the Heat Capacity control center, (b) select **Switch to New** in the **Files** tab in the Heat Capacity control center, or (c) select **Change** in the **Current Calibration File** panel included in the addenda and sample measurement puck installation wizards.
2. If you are not editing the title of the active calibration, click once on the row that is part of the .cal file you want to edit. The row is highlighted, but the two arrows do not move to the left-most row column.
3. Select **Edit Title**. Then edit or enter the title as necessary, and select **OK**. The edits appear in the appropriate **Title** column in the **Calorimeter Files** dialog box. If you have edited the title of a nonactive file, the highlight bar returns to the active file.
4. Select **OK** to close the **Calorimeter Files** dialog box.

5.7.3 Deleting a Calibration File

You may delete any .cal file that is not the active calibration. Once you issue the **Delete** command, the .cal file is deleted permanently and may not be retrieved.

Complete the following steps to delete a .cal file:

1. Open the **Calorimeter Files** dialog box (Figure 5-1 or 5-7) by doing one of the following: (a) double-click on the **Calibration** status panel in the Heat Capacity control center, (b) select **Switch to New** in the **Files** tab in the Heat Capacity control center, or (c) select **Change** in the **Current Calibration File** panel included in the addenda and sample measurement puck installation wizards.
2. Click once on any row that is part of a nonactive .cal file. The row is highlighted, but the two arrows do not move to the left-most row column.
3. Select **Delete**, and then select **Yes**. The file name is deleted immediately, and the highlight bar returns to the active file.
4. Select **OK** to close the **Calorimeter Files** dialog box.

5.7.4 Advanced Calibration File Manipulation

By clicking the **Advanced** button in the **Calorimeter Files** dialog there are several commands available for manipulation of .cal file data. These commands must first be enabled by selecting **Enable Advanced Table Operations** from the popup menu.

To view the complete interpolated platform thermometer calibration in a single curve covering the whole temperature range, select the **Advanced >> Export Interpolated Curve from Joined Table** item. This command will ask for a magnetic field at which to extract the calibration curve. The resulting curve will be displayed in a MultiVu graph window. Since the calculated specific heat at a

given temperature is proportional to the slope of this curve, the slope of resistance versus temperature is also displayed. Bumps or other features in this curve may indicate a flawed calibration.

The table conditioning algorithms that are run at the end of pass 1 of the calibration procedure (see section 5.6.1) can be applied manually from the **Advance** menu by selecting **Advanced >> Create Standard Smoothed Version of Active Calibration File**. This procedure will remove kinks and other defects in the calibration data. This is particularly useful when applied to calibration files that were calibrated with an earlier version of the software. It may also be useful if individual tables have been manually edited as described below. Statistical information about the transformation is also generated as part of this operation and is written to a detailed comparison data file for viewing in MultiVu.

Although not recommended, manipulation of individual calibration tables can be performed by opening one of the individual tables as described in Section 5.5. To change a table entry, open the **Calibration Table Edit** dialog (for example, as shown in Fig. 5-8) for the desired table. Then, select the **Edit** tab, change an entry in the text box, then click the **Parse** button. When you are finished, click the **OK** button. This technique can also be used to manipulate addenda tables. See Chapter 6 for more information on addenda tables.

After performing any operations on the calibration file, including editing of the individual tables, you can save or discard the results using the **Advanced >> Save Active Calibration** commands or the **Advanced >> Discard Changes and Reload Calibration** command from the **Calorimeter Files** dialog.

Creating and Using Addenda Tables

6.1 Introduction

This chapter contains the following information:

- Section 6.2 presents an overview of addenda measurement.
- Section 6.3 explains how to measure the addenda.
- Section 6.4 summarizes the addenda parameters.
- Section 6.5 describes the tables for addenda measurement.
- Section 6.6 describes the addenda measurement process.
- Section 6.7 discusses addenda table management.

6.2 Overview of Addenda Measurement

The addenda measurement is the measurement of the heat capacity of the sample platform. The Heat Capacity software calculates the heat capacity of a sample by subtracting the addenda measurement from the total heat capacity measurement. The two measurements—one with and one without a sample on the sample platform—are necessary. In the presence of a magnetic field, it may also be necessary to subtract addenda measurements obtained at each field.

For accurate measurement of the sample heat capacity, you first apply a small amount of grease—just enough to hold the sample—to the sample platform, and then you measure the heat capacity of the grease and the platform: This is the addenda measurement. Next, you mount the sample on the sample platform by pressing the sample onto the grease you have already applied to the platform. You then measure the heat capacity of the sample as described in Chapter 7. Automatic subtraction of the addenda, interpolated at each sample measurement temperature, is performed.

Because each addenda measurement is characteristic of the puck, the Heat Capacity software stores a puck's addenda data in the active calibration, or `.cal`, file. The software creates a new addenda table for each addenda measurement.

6.2.1 Addenda Tables

Addenda tables are stored in the `.cal` file. The software creates a new addenda table each time you measure the addenda; one addenda table summarizes the addenda data for one measurement. You may take any number of addenda measurements for a puck, and the `.cal` file may store any number of addenda tables. Only one addenda table is active at a time. By default, the software initializes with the last active addenda table for a given `.cal` file. You may select any addenda table (see Section 6.7.1) within the active `.cal` file in order to make that addenda table active.

6.2.2 Active Addenda Table

The **Addenda** status panel in the Heat Capacity control center indicates the active addenda table. The panel displays the date that the addenda was measured and the temperature range of the measurement. When you run another addenda or select a different addenda table, the creation date and temperature range of that addenda table appear in the status panel.

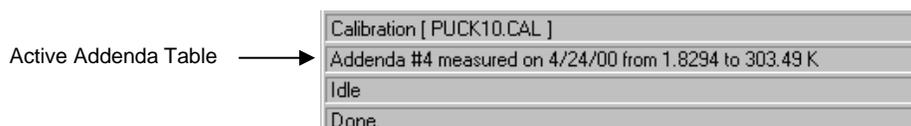


Figure 6-1. Active Addenda Table Identified in **Addenda** Status Panel in Control Center

6.2.3 Use of Data Files

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

6.2.4 Use of Grease

Proper thermal contact between the sample and sample platform requires that grease be used. For accurate measurement of a sample's heat capacity, the grease must be included as part of the addenda. Therefore, you apply grease to the sample platform before you measure the addenda. Use only the amount of grease necessary to attach the sample to the sample platform. The sample's size and geometry determine the correct amount of grease. Generally, one cubic millimeter should be sufficient. If you use too much grease, the poor thermal diffusivity of the grease may yield a poor measurement of the addenda, thus resulting in an inaccurate sample heat capacity measurement. To ensure that the addenda is properly measured, spread the grease out, on the sample platform, in a thin layer over an area that matches the contact area with the sample.

Care is also required during sample mounting to preserve the exact amount of grease present during the addenda measurement. Any discrepancy between the amounts of grease used for the addenda and sample measurements affects the accuracy of the sample heat capacity measurement.

6.2.4.1 RECOMMENDED GREASES

Quantum Design recommends either Apiezon N Grease or Apiezon H Grease¹ for use as an adhesive to attach most samples to the sample platform. Each type of grease has advantages and disadvantages that mainly depend on the range of temperatures over which it will be used.

The consistency of N Grease at room temperature makes it easy to attach samples to the platform and convenient to remove samples from the platform. However, between 260 K and 325 K, N Grease exhibits a slight anomaly in its specific heat near its melting point. This has been documented in the literature (see Bunting, et al, Cryogenics, Oct. 1969, pp. 385-386). The heat capacity of N Grease is only reproducible to about 5% in the temperature range 260 K to 325 K. Also, the heat capacity of the grease exhibits peaks in temperature in this range. For best results, use N grease only at temperatures less than 220 K.

Apiezon H Grease has good properties for higher temperature measurements. Since it doesn't melt like N Grease, its specific heat is smooth and reproducible. However, it is more difficult to handle at room temperature, because it does not "wet" the surfaces of the platform or sample very well. Also, at temperatures below 200 K, H Grease has a tendency to spontaneously pop off. So for best results, use H grease for measurements above 200K. For information on the heat capacity of both N and H Grease, refer to "Heat Capacity of Apiezon H Grease from 1 to 50K" by A. J. Bevolo (Bevolo 1974).

If you need to work over the entire temperature range, N Grease is probably the best choice. Although smoothness and accuracy will be worse than with H Grease in the range of the anomaly (260 K to 325 K), overall, the results will be better than H Grease since there will be no loss of grease from it popping off. Improved results can be obtained with N Grease by using the smallest possible amount of grease to hold the sample. Also, the addenda can be sampled densely enough in the range of the anomaly so that the peaks are resolved and can be reasonably subtracted when the sample measurement is performed. An addenda measurement sampled at 5 K intervals between 200 K and 325 K is adequate to eliminate this under-sampling problem.

To summarize:

- Use N Grease at temperatures < 220 K
- Use H Grease at temperatures > 200 K
- For measurements over the entire temperature range, Use N Grease and refer to the above discussion.

6.2.5 Magnetic Field Dependent Addenda

Due to the materials used in the construction of the heat capacity platform, the heat capacity of the addenda depends on magnetic field. The effect is significant only at temperatures below about 10 K, but it is quite large for the original version of the heat capacity platform (see Table 6-1). This effect has been greatly reduced in the current version of the heat capacity platform. You can distinguish between platform versions by their appearance: the original version is opaque white and the current version is translucent (see-through).

¹ Apiezon N Grease and Apiezon H Grease are produced by Apiezon Products Ltd., London, England.

Table 6-1. Comparison between Original and Current Heat Capacity Pucks

Temperature (K)	Original Design		Current Design		
	Addenda at zero field (nJ/K)	Max. addenda change when applying field ² (nJ/K)	Standard puck addenda in zero field (nJ/K)	He-3 puck addenda in zero field (nJ/K)	Max. addenda change when applying field ¹ (nJ/K)
0.4	130	125	N/A	5	1.5
0.6	130	120	N/A	7	2
1	120	100	N/A	15	2
1.5	120	70	N/A	25	2
2.5	180	10	155	90	2
4	470	120	470	310	NM
6	1350	200	1350	1000	NM
10	5400	300	5400	4000	NM

¹ "Maximum addenda change when applying field" is the absolute value of the maximum difference between $C(H)$ and $C(0)$ for the range $0 < H < 16$ T. Note that the maximum difference does not necessarily occur at the maximum magnetic field.

² NM means not measurable within the instrument noise.

To obtain meaningful heat capacity data in magnetic fields when measuring with temperatures below 10 K, you might find it necessary to measure the addenda contribution at the specific fields of interest so that the heat capacity software can perform an accurate addenda subtraction during sample heat capacity measurements. It is especially important to follow this procedure if you are using pucks of the original design, but it usually is not necessary with pucks of the new design. The field dependence of the latter is small enough that you will rarely need to perform field-dependent addenda measurements when you work at temperatures above 1.9 K, and you usually will not need to perform field-dependent addenda measurements when you work at temperatures below 1.9 K.

Rather than measuring the addenda heat capacity in all magnetic fields of interest, you could first measure the addenda only in zero (0) field. If you choose this approach, you should compare the field dependence of your measured sample heat capacity to that of the addenda heat capacity to ensure that the field dependence of the addenda has not significantly affected your data. From your data, calculate the measured change in the sample heat capacity when applying a magnetic field at a few representative temperatures. Compare these values to those in Table 6-1 for your version of the heat capacity puck (original or current version). If the changes in Table 6-1 are comparable to or greater than the change you calculated in the sample heat capacity, then the sample heat capacity data in magnetic field might suffer significant inaccuracy due to the addenda field dependence. Under the latter circumstances, you should perform addenda measurements in field in order to achieve accurate sample heat capacity in a magnetic field.

If you determine that you must perform addenda measurements at each field of interest, be sure to use the appropriate addenda table for each field when you measure sample heat capacity. You can select addenda tables manually or in a sequence, as explained in Sections 6.7.1 and 6.7.2.

6.3 Measuring the Addenda

6.3.1 Apply Grease to the Sample Platform

1. Plug in the small vacuum pump that is supplied with the sample-mounting station. Verify that the sample-mounting station is receiving vacuum.
2. Slide the thermal radiation shield off the puck if the shield covers the puck.
3. Place the puck, with the sample platform facing upward, inside the sample-mounting station's puck holder. Rotate the puck until the index key slips into the notch at the rear of the holder, and then *gently* push the puck into the holder. Verify that the sample platform is properly seated on the platform holder.
4. Pivot the puck interlock arm toward the puck so that it grabs and immobilizes the puck. When the interlock arm is against the puck, vacuum pulls the sample platform downward.

A hissing sound in the puck holder indicates a poor seal between the sample platform and holder. To eliminate the leak, remove the puck from the holder, clean off all debris that is under the puck, and then place the puck back inside the holder.

5. Position the sample-mounting station and the puck below a wide field stereo microscope and a strong light, if desired.

CAUTION!

Caution and a steady hand are required to mount and remove samples. You risk breaking the puck wires unless you work very carefully *and* you feel comfortable using tweezers to manipulate samples under the microscope. If you break a wire, you must replace the entire puck frame and platform along with the wires. Section 10.4.1 explains this procedure.

6. If a sample from a previous measurement is mounted on the sample platform, remove that sample. Use tweezers to gently slide the sample off the platform. Never pull the sample directly up off the platform. Pulling the sample directly upward may pull the platform off its mount and break the wires.
7. Use a toothpick or the broken edge of a cotton-tipped applicator to apply a small amount of grease to the center of the sample platform. *Apply only enough grease to attach the sample to the platform.* Make certain that grease does not extend over the edges of the platform or touch the wires. Section 6.2.4.1 describes recommended greases. (But do not leave behind cotton fibers).
8. Use a cotton-tipped applicator to clean superfluous grease off the platform. Start in the center of the platform, and brush the grease outwards. Make certain that grease does not drip over the edges of the platform or touch the wires.
9. Push the puck interlock arm away from the puck.
10. Pull the puck off the puck holder.
11. Hold the puck so that the sample platform is facing upward. Slide the thermal radiation shield over the top of the puck, and then twist the shield to verify that it is securely in position. (See warning in Section 11.3.6).

Note: Use the puck adjustment tool (see Section 9.2) on the puck after the puck has been inserted in the sample chamber approximately 10 times. By adjusting the tension in the chuck fingers, the puck adjustment tool ensures solid thermal contact between the puck and the heater block located at the bottom of the sample chamber.

12. Use the broken edge of a cotton-tipped applicator or a toothpick to apply a small amount of Apiezon H Grease to each chuck finger (Figure 3.1) if the fingers are dry. Apply only enough H Grease to make the fingers slightly sticky. Make certain that H Grease does not drip between the fingers.
13. Unplug the vacuum pump if you will not immediately reuse the sample-mounting station.

6.3.2 Insert the Puck

1. Locate the puck's serial number, which is written on the green fiberglass connector at the base of the puck (see Figure 3-2). You need to know the serial number to select the `.cal` file that has been created for the puck.
2. Select the **Installation Wizards** tab in the Heat Capacity control center.
3. Select **Prepare Addenda Measurement**. The **Installing Puck for Addenda Measurement** dialog box opens.
4. Select **Open Chamber** to vent the sample chamber and warm it to room temperature.

5. Insert the puck into the sample chamber when the on-screen instructions prompt you to do so. The *Physical Property Measurement System: Hardware Manual* discusses puck insertion in detail.
6. Install the baffle assembly. Verify that the charcoal holder is attached to the bottom of the assembly. Refer to Figures 3-7 and 3-8B.

CAUTION!

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

7. Insert the baffle assembly into the sample chamber.
8. Select **Purge** to purge and seal the sample chamber.
9. Select **Next**. Then enter the puck's serial number.
10. Select **Next** to open the **Current Calibration File** panel.

6.3.3 Select a Calibration File

Review the information identifying the active .cal file in the **Current Calibration File** panel (see Figure 6-2). The panel displays summary information—serial number, creation date, name, title, temperature range, and the number of associated addenda tables—for the active .cal file. The serial number identified in the **Current Calibration File** panel should match the serial number you entered previously. A warning message appears at the bottom of the panel if the two numbers do not match.

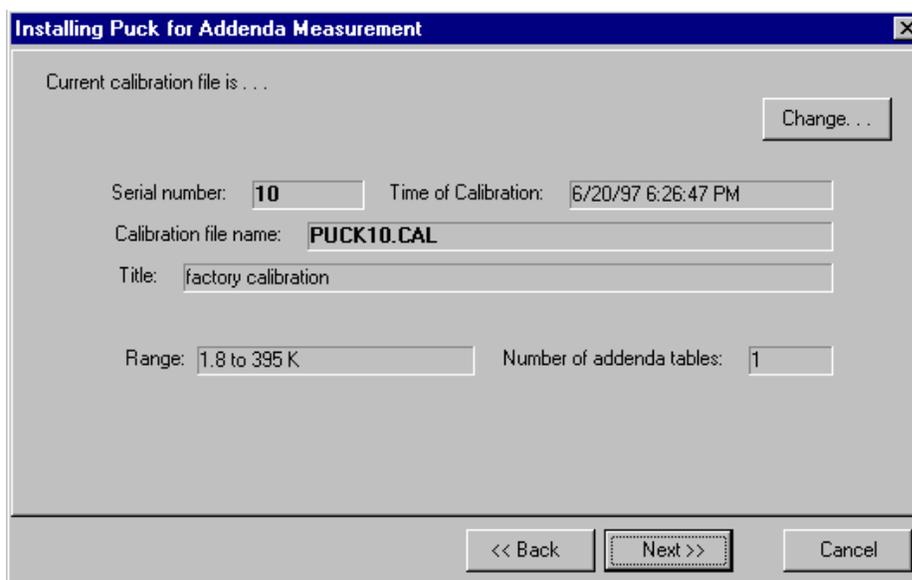


Figure 6-2. **Current Calibration File** Panel in Puck Installation Wizard

- If the serial numbers match, indicating that the correct .cal file is active, select **Next** to open the **Puck Test Results** panel.
- If the serial numbers do *not* match, indicating that the correct .cal file is not selected, you must select the correct .cal file. Do the following:
 1. Select **Change** in the **Current Calibration File** panel. The **Calorimeter Files** dialog box opens (see Figure 5-1 or 5-7).
 2. Locate the .cal file that includes the puck's serial number. The serial number appears in the name of the file and in the **Ser#** column. If necessary, adjust the size of the columns or use the scroll bar to view all information in the columns.
 3. Select the correct .cal file as follows: (a) click anywhere on the row that summarizes the data saved in the correct .cal file, (b) click on **Select**, and (c) click on **OK** in the pop-up message. The file is selected only when the row is highlighted and the two arrows appear to the left of it.
 4. Select **OK** in the **Calorimeter Files** dialog box. The **Current Calibration File** panel now displays all data stored in the .cal file you have activated. Any warning message disappears if the serial numbers match.
 5. Select **Next** to open the **Puck Test Results** panel.

6.3.4 Test the Puck

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

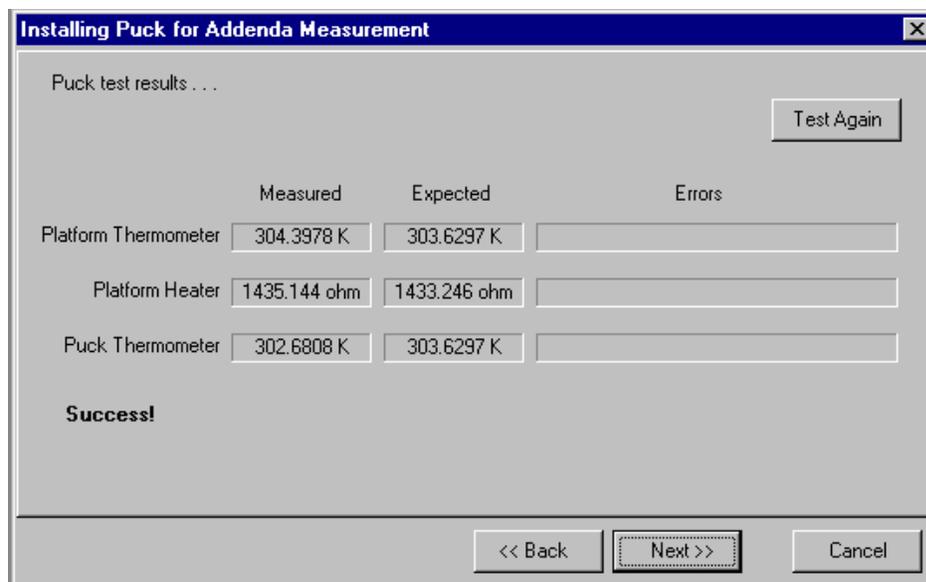


Figure 6-3. **Puck Test Results** panel in puck installation wizard. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

A failure might indicate a broken puck wire or a loose or unplugged cable or that the temperature of the puck is changing too rapidly. If a puck wire is broken, you must replace the puck frame. Refer to Section 9.4.1.

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

6.3.5 Select a Data File

Note: Opening a data file to save the addenda measurement data is optional. However, if you want to plot the addenda data, you must save it to a data file. You may use the currently selected file, create a new file, or append data to an existing file.

Note: In certain cases, it might be useful to append new measurements to the end of an existing file. When you append to an existing file, the original header information and the measurement units that were originally selected are preserved in the file.

Review the **Header Information** in the **Data File Name** panel. The **Header Information** lists the information stored in the header of the active data file. The **Header Information** is blank if a data file is not open.

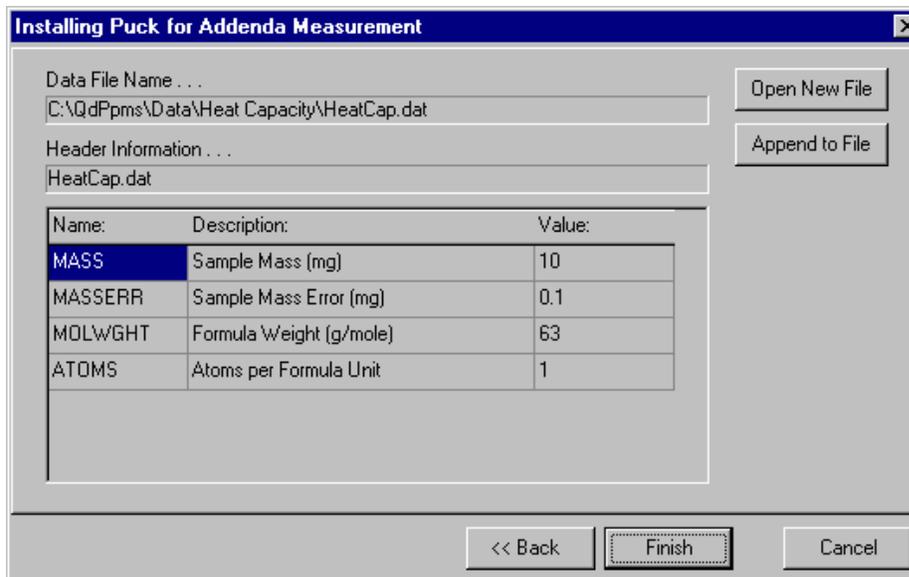


Figure 6-4. **Data File Name** panel in puck installation wizard. The name and location of the active data file are identified at the top of the panel. The information in the header of the active file is displayed below the **Header Information** box. If no data file is active, the **Data File Name** and **Header Information** boxes are blank.

- If you want to use the data file that is currently active, select **Finish**. The **Data File Name** panel closes.
- If you want to append data to a different data file, select **Append to File**, select the file, and then select **Open**. The **Header Information** in the **Data File Name** panel lists the information stored in the header of the file you have just selected. Select **Finish** to close the **Data File Name** panel.
- If you want to create a new data file, do the following:
 1. Select **Open New File** in the **Data File Name** panel.

2. Enter the name of the file, and then select **Save**. If you have entered the name of an existing file, a pop-up message asks whether you want to replace the existing file. Select **No**, and then enter another file name.
Once you save the name of the new data file, the **New Data File Information** dialog box opens. The **Optional Information** displayed in this dialog box is the user-defined information that will be saved to the data file header.
3. Enter a different title for the graph view of the file, if necessary. By default, the file name is also the title of the graph view.
4. Review the user-defined **Optional Information** that will be saved to the file header. Select **Configure List** to add, edit, or delete optional items. See Section 4.7.2.1 for more information.
When you create a new data file to store addenda measurement data, you are not prompted to define any sample information that will be saved to the data file header. The software ignores sample information when it measures the addenda.
5. Select **OK** to close the **New Data File Information** dialog box. The **Data File Name** panel appears again. The **Header Information** now displays the header of the data file you have just opened.
6. Select **Finish** to close the **Data File Name** panel.

6.3.6 Run the Addenda Measurement

1. If you will measure addenda in a magnetic field, you must first set the magnet persistent at the desired field. When the addenda measurement begins, the current field will be recorded for subsequent heat capacity measurements.
2. Select the **Measurement** tab in the Heat Capacity control center.
3. Select **Create New Addenda Table**. The **Create New Addenda Heat Capacity Table** dialog box opens. You use this dialog box to specify the parameters for the addenda measurement. The dialog box displays the values used for the last addenda measurement. The parameters are organized into a **Setup** tab (Figure 6-5) and an **Advanced** tab (Figure 6-6). Section 6.4 explains the parameters in more detail.

Note: Under certain circumstances, residual helium may be absorbed by components of the calorimeter if the calorimeter is left below 6 K for several hours prior to a measurement. If this is the case, it is recommended that the system be thermally cycled to above 6 K prior to a measurement or that you measure with decreasing temperatures starting above 6 K.

4. Select the **Setup** tab. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

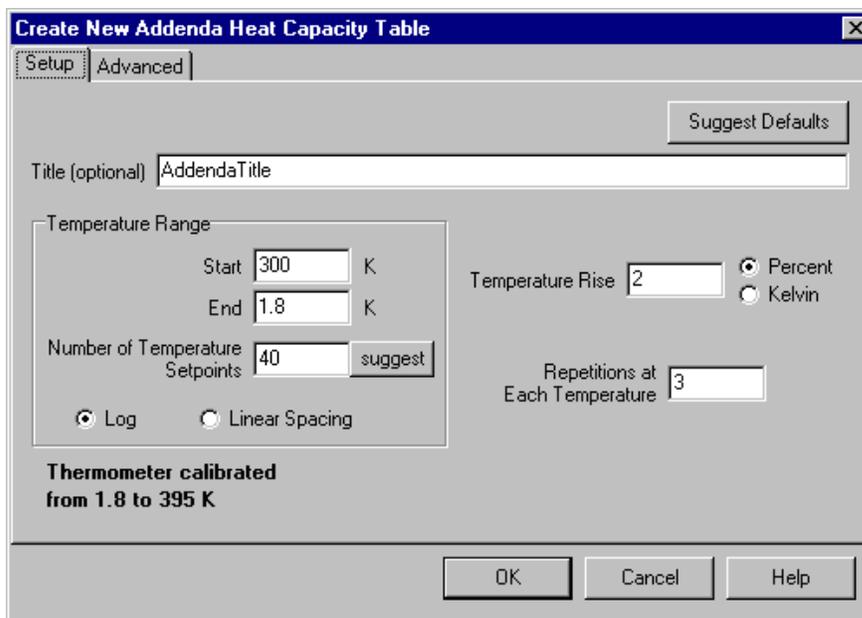


Figure 6-5. Setup Tab in Create New Addenda Heat Capacity Table Dialog Box

5. Enter the title of the addenda table. Make the title as descriptive as possible so that you can match the addenda table with the correct sample.
6. Enter the starting value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range, which are displayed in the lower left corner of the dialog box. The first measurement is taken at the starting temperature value.
7. Enter the ending value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range. The last measurement is taken at the ending temperature value.
8. Enter the number of temperature values, including the starting and ending temperature values, for which you want to take measurements. Pressing the **suggest** button inserts a value large enough to ensure good interpolation, yet small enough to avoid unnecessary over-sampling. See Section 13.3.3 for special considerations when using N grease in the 200K to 300K range.

Note: Selecting exactly matching measurement points for the sample heat capacity measurement and the addenda measurement is unnecessary. During the sample heat capacity measurement, the software uses polynomial interpolation of the active addenda table at the measurement temperatures for determining the appropriate addenda to subtract.

9. Select the type of spacing that is used to separate the temperature values.
10. Enter a value for the temperature rise.
11. Enter the number of times a measurement is repeated at each temperature.
12. Select the **Advanced** tab if you want to review the advanced measurement parameters. Quantum Design recommends that only default advanced measurement parameters be used. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

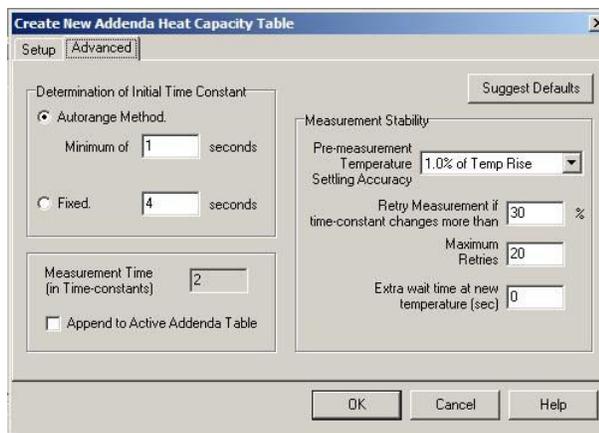


Figure 6-6. **Advanced** Tab in **Create New Addenda Heat Capacity Table** Dialog Box

13. Select the **OK** button at the bottom of the **Create New Addenda Heat Capacity Table** dialog. The addenda measurement begins if the temperature range you defined is within the calibrated temperature range. If the temperature range is outside the calibrated range, a warning message that lists the suggested values pops up. You may override the warning and run the addenda.

As soon as the addenda begins to run, the Measurement Status Viewer opens to show the measurement's progress. The name of each task that is part of the measurement appears, as it is performed, in the message list box at the bottom of the Viewer. When the software performs a new measurement, it presents the data in the measurement-field panels and plots the data as a graph. Section 4.5 discusses the Measurement Status Viewer in detail.

The bottom status panel in the Heat Capacity control center also indicates the name of each task as the task is performed.

Section 6.6 describes the addenda measurement process in more detail.

14. Wait for the measurement to finish. A measurement generally lasts several hours. When the measurement is complete, the **Idle** and **Done** messages appear in the bottom status panels in the Heat Capacity control center.

6.3.6.1 PAUSING OR ABORTING A MEASUREMENT

You may pause and resume an addenda measurement or abort an addenda measurement at any time.

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

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

6.3.7 Remove the Puck

Refer to the *Physical Property Measurement System: Hardware Manual* to remove the puck from the sample chamber.

6.4 Summary of Addenda Parameters

Table 6-2. Parameters in **Setup** Tab in **Create New Addenda Heat Capacity Table** Dialog Box

PARAMETER	DEFINITION
Title	Descriptive title for identifying this addenda in Addenda Tables dialog.
Temperature Range Starting Value	Starting value for temperature range. First measurement is taken at this temperature. Addenda temperature range should be within temperature range of thermometer calibration.
Temperature Range Ending Value	Ending value for temperature range. Last measurement is taken at this temperature. Addenda temperature range should be within temperature range of thermometer calibration.
Number of Temperature Values	Number of temperature values, including starting and ending value, at which measurements are taken. Pressing suggest button inserts a value large enough to ensure good interpolation, yet small enough to avoid unnecessary over-sampling.
Spacing for Temperature Values	Logarithmic or linear spacing between temperature values. Logarithmic spacing equally spaces logarithms of temperature values. Linear spacing equally spaces temperature values.
Temperature Rise	<p>Temperature rise produced by heater-on cycle of heat capacity measurement. Large temperature rise reduces scatter. Small temperature rise is necessary if features such as peaks must be resolved. While there should be no such features in the addenda itself, it is a good idea to use the same temperature rise for the addenda and subsequent heat capacity measurement. A typical value for temperature rise is 2%.</p> <p>Temperature rise is either given in kelvin or as a percentage of sample temperature at each measurement, depending on user selection. The application automatically determines the heater current needed to produce this temperature rise.</p>
Number of Repetitions Per Measurement	Number of times a measurement is repeated at each temperature. A value of two or more is recommended since this gives good indication of self-consistency of measurements.

Table 6-3. Parameters in **Advanced** Tab in **Create New Addenda Heat Capacity Table** Dialog Box

PARAMETER	DEFINITION
Determination of First Time Constant: Autorange Method	If this option is selected, initial time constant is determined by applying a constant heater power and determining amount of time it takes for temperature to rise to half the requested delta. The minimum value is used if the resultant time constant is less.
Determination of First Time Constant: Fixed	If this option is selected, initial time constant is the specified value. Use this if you already roughly know (less than a factor of two or so) the time constant from previous measurements at this temperature. The auto ranging step will be skipped, thus obtaining first measurement more quickly.
Relaxation Duration: Measurement Time (In Time Constants)	Amount of time, including heater-on and heater-off sequences, used for a single relaxation measurement. For “standard” puck type, this value is always 2.
Append to Active Addenda Table	When selected, the addenda measurements are appended to current addenda table instead of creating a new table. Only temperatures that extend (above or below) the existing table are allowed. This is useful for continuing an aborted run, or for creating non-uniform addenda spacing. (For example, see section 12.3.3.)
Measurement Stability: Settling Accuracy	Determines how long software waits for temperature stability before taking each measurement. Settling accuracy is given as a percentage of temperature rise. If sample-platform temperature changes between successive readings by less than the settling-accuracy value, temperature is stable enough to proceed with measurement. Small settling accuracies lead to better temperature reproducibility at a specific temperature, though not necessarily better heat capacity values. That is, the temperature scatter will be smaller when this value is smaller but heat capacity values will still fall on correct curve. However, the measurement will take longer with a smaller value. A typical value for settling accuracy is 1%.
Measurement Stability: Retry Percentage	Determines how much the time constant is allowed to change from measurement to measurement. For example, a typical value of 30% would mean that if previous relaxation measurement had a time constant of 1 second, and the current measurement was greater than 1.3 or less than 0.7, the current measurement would be discarded and the measurement repeated with the latest acquired time constant. A smaller value may reduce temperature hysteresis effects with some samples by requiring better self-consistency between the assumed time constant used for the “Measurement Time” and the actual time constant determined from the fit.
Measurement Stability: Maximum Retries	Maximum number of times the above condition will discard a measurement before doubling the user-specified “Measurement Time.” This is an attempt to recover self-consistency. The assumption is that the sample is possibly very poorly attached or that specified Measurement Time is much less than 1 and is too short to discern a good value for the time constant when the data is fit. All subsequent measurements will be performed with the longer time constant.
Measurement Stability: Extra wait time at new temperature (sec)	After the PPMS declares stability at a new temperature, the software will wait for the indicated time before starting the measurement. Subsequent measurements at the same temperature do not wait.

6.5 Description of Addenda Tables

The addenda table for any addenda measurement contains two separate tables—one displaying the heat capacity values of the sample platform, and one displaying error bar values—plus an optional user-defined offset table. When the Heat Capacity software measures the heat capacity of a sample, it determines the errors from the fitting algorithm. The offset table, which is read from a separate file in the TempCal directory, is used in cases where a known heat capacity from, for example, an auxiliary sample holder, should also be included in the addenda. Section 6.7.3 discusses offset tables in more detail.

You view any addenda table by selecting the appropriate command button in the **Addenda Tables** dialog box. You open the **Addenda Tables** dialog box (Figure 6-9) by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.

- To view the table of addenda heat capacity values versus temperature, select **Addenda Entries** in the **Addenda Tables** dialog box.
- To view the error table, select **Addenda Error Entries** in the **Addenda Tables** dialog box.
- To create the optional user-defined offset table, select **Addenda Offset Formula** in the **Addenda Tables** dialog box.

The screenshot shows a dialog box titled "Calibration Table Edit" with a menu bar containing "File". Below the menu bar is a table with two columns: "Temp" and "AddendaHC". The table contains six rows of data. Below the table is a "Calculator" section with two input fields labeled "Temp" and "AddendaHC", and two buttons: "==" and "<==". Below the calculator are "OK" and "Cancel" buttons.

	Temp	AddendaHC
1	1.829363	0.01001167
2	2.082635	0.0148385
3	2.371341	0.02199852
4	2.701166	0.03260193
5	3.075822	0.04831461
6	3.503663	0.07162027

Figure 6-7. Table Displaying Addenda Entries

The screenshot shows a dialog box titled "Calibration Table Edit" with a menu bar containing "File". Below the menu bar is a table with two columns: "Temp" and "AddendaHCErr". The table contains six rows of data. Below the table is a "Calculator" section with two input fields labeled "Temp" and "AddendaHCErr", and two buttons: "==" and "<==". Below the calculator are "OK" and "Cancel" buttons.

	Temp	AddendaHCErr
1	1.829363	4.337284E-6
2	2.082636	5.974088E-6
3	2.371341	1.561693E-5
4	2.701166	2.675387E-5
5	3.075822	2.857165E-5
6	3.503663	0.0001165942

Figure 6-8. Table Displaying Addenda Errors

6.6 Description of Addenda Measurement Process

The Heat Capacity software uses similar procedures to acquire data for addenda measurements and sample heat capacity measurements. The main differences between the two types of measurements are how the software converts raw data to a heat capacity value and how the software saves heat capacity data. Section 7.6 describes the sample heat capacity measurement process.

When an addenda measurement begins, the vacuum system first evacuates the sample chamber. Then the chamber temperature stabilizes at the user-defined starting temperature value. Measurements begin when the temperature is stable. The software measures the temperature response of the sample platform to the applied heat, and then fits the acquired time trace to a model that assumes a sample is not present; that is, a simple model (Section 4.3.1.1) is used for the fit. The use of this model is an important difference between the analysis of the data for determining the addenda heat capacity and the sample heat capacity.

Once the software has determined the heat capacity value and the associated error bar, it writes these values to a new addenda table residing in the active `.cal` file. A new addenda table is created each time you run the procedure. You can append data to an existing table if you select the **Append to Active Addenda Table** option on the **Advanced** page as described in Table 6-3. The current magnetic field value is also written to the `.cal` file and appears in the **Addenda Tables** dialog box. The software never automatically overwrites an addenda table and deletes a table only when you specify that it do so. Section 6.7.5 explains how to delete an addenda table.

If a data file is open before the addenda measurement begins, the software writes all fit information and the heat capacity of the addenda to the data file. However, when the software measures the heat capacity of a sample, it uses only the information written to the addenda table to compute the sample heat capacity.

6.7 Managing Addenda Tables

The software saves the addenda table inside the active `.cal` file. The **Addenda Tables** dialog box conveniently displays all addenda tables created for the active `.cal` file. Each horizontal row in the dialog box summarizes the data in one addenda table.

You open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.

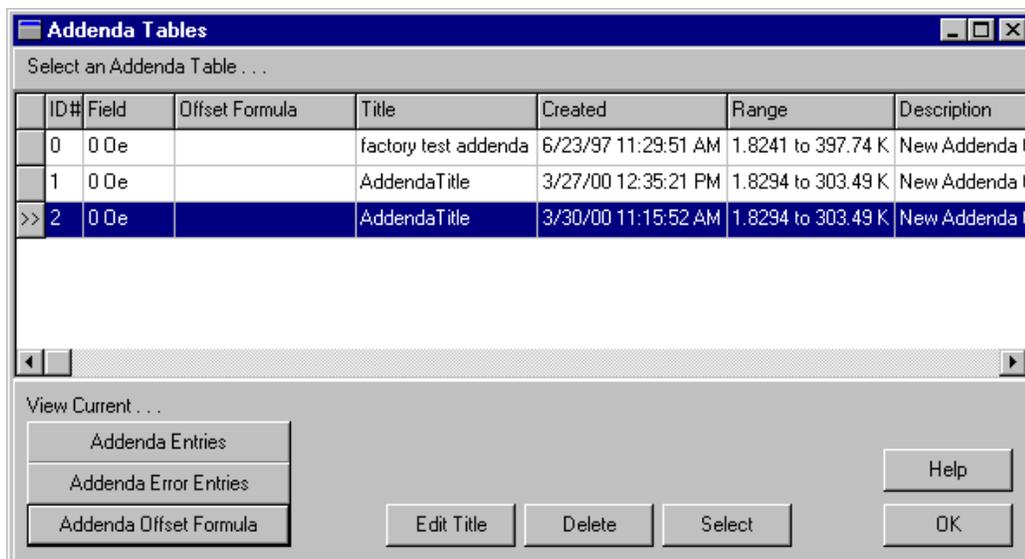


Figure 6-9. **Addenda Tables** Dialog Box

A .cal file may include any number of addenda tables, although only one addenda table is active at a time. The **Addenda Tables** dialog box indicates which addenda table is active. You may select any other addenda table to make it active.

The magnetic field at which the addenda was acquired is included as part of the description. Also, each addenda has a unique **ID number**. Both these fields can be used for identifying the addenda table in the **Switch Addenda** sequence command as described in Section 6.7.2.

Note: Superfluous addenda tables should periodically be deleted (see Section 6.7.5). A large number of addenda tables may overload the system’s memory and impede system performance.

6.7.1 Selecting an Addenda Table Manually

Only one addenda table is active at a time and remains active until a new addenda measurement is performed or a different table is selected. You complete the following steps to select an addenda table:

1. Verify that the correct .cal file is active. Refer to Section 5.7.1. A .cal file contains only the addenda measurements taken for one puck.
2. Open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.

If necessary, adjust the size of the table columns in the **Addenda Tables** dialog box or use the scroll bar to view all information in the columns.

3. Select the correct addenda table as follows: (a) click anywhere on the row that summarizes the data saved in the correct addenda table, (b) click on **Select**, and then (c) click on **OK** in the pop-up message. The entire row is highlighted, and then two arrows move to the left-most column of the row. The addenda table is selected only when the arrows appear to the left of it.
4. Select **OK** to close the **Addenda Tables** dialog box. If necessary, select **OK** to close the **Calorimeter Files** dialog box. The **Addenda** status panel in the Heat Capacity control center now indicates the creation date and temperature range of the addenda table you just selected.

6.7.2 Switching to a New Addenda Table from a Sequence

Switching to a new addenda table under sequence control may be necessary when measuring heat capacity in magnetic fields. Since the heat capacity of the sample holder is somewhat field dependent (see section 6.2.5), automatic addenda switching enables fully automated heat capacity measurements at different fields.

To switch to a new addenda table, simply include the **Switch Addenda** command in your sequence. You can use the command to switch to a new addenda table any one of three different ways.

- **By title.** When selecting by title, you supply a substring contained in the title of the addenda. This is the same title specified in the **Create New Addenda Heat Capacity Table** as shown in Figure 6-5 and in the **Addenda Tables** dialog as shown in Figure 6-9. When more than one addenda table in the current `.cal` file is a match, the most recently acquired table is selected.
- **By magnetic field.** When selecting by magnetic field, the table with the closest magnetic field is selected. Again, in the case of more than one match, the most recently acquired table is selected.
- **By ID number.** When selecting by ID number the specified table is selected. Select the **Browse** button to bring up a list of currently defined addenda tables and associated ID numbers. ID numbers are automatically selected for each addenda table when it is first created.

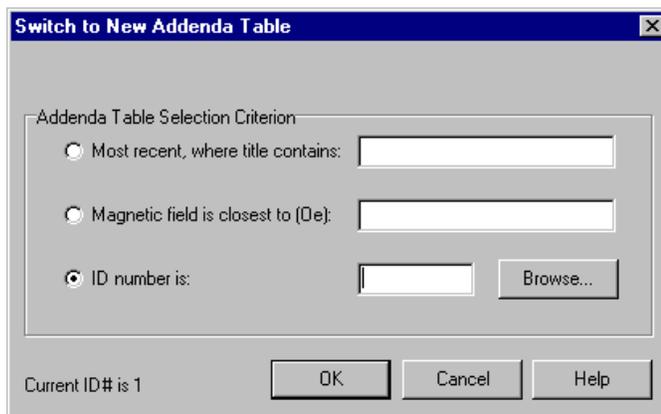


Figure 6-10. **Switch Addenda** Sequence Command Dialog Box

6.7.3 Optional Addenda Offset Tables

In some cases it is useful to subtract a known heat capacity dependence from the one already contained in the addenda table. For example, if your sample is in a container of known mass, it is necessary to subtract the heat capacity of the container to obtain the heat capacity of the contained sample. If the heat capacity of the container has already been measured as a function of temperature, you can simply include that data as part of the addenda.

An offset file must be placed in the `TempCal` directory and must have a `.off` suffix in the file name. The easiest way to create such a file in the proper format is to specify the name of the offset data in the **Advanced** tab of the **Heat Capacity Versus Temperature** dialog box when you are measuring the container. A scale factor can be specified when the table is used.

To specify an offset file for a specific addenda table after the addenda table has been measured, select the **Addenda Offset Formula** button in the **Addenda Tables** dialog box. Then use the **Addenda Offset Formula** dialog box (Figure 6-11) to specify contributions from up to two offset files, each with its own scale factor applied.

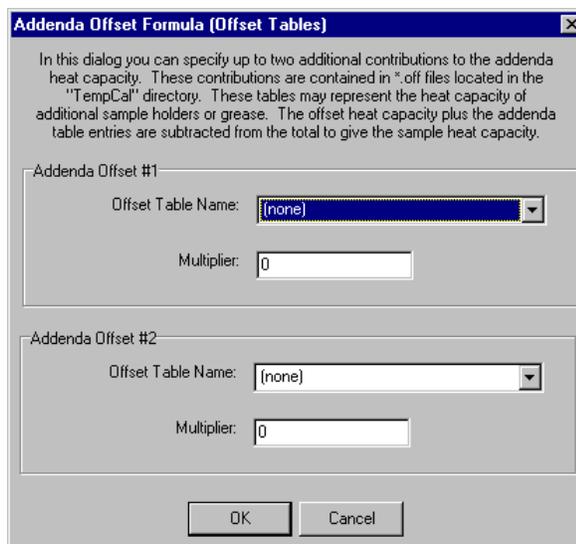


Figure 6-11. Addenda Offset Formula Dialog Box

6.7.4 Editing the Title of an Addenda Table

If a `.cal` file includes several addenda tables, you should assign descriptive titles to the tables to help distinguish them. You may edit the title of an addenda table at any time; the table does not need to be active.

Complete the following steps to edit the title of an addenda table:

1. Verify that the correct `.cal` file is active. Refer to Section 5.7.1. A `.cal` file contains only the addenda measurements taken for one puck.
2. Open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.
3. If you are not editing the title of the active addenda table, click once on the row that is part of the addenda table you want to edit. The row is highlighted, but the two arrows do not move to the left-most row column.
4. Select **Edit Title**. Then edit or enter the title as necessary, and select **OK**. The edits appear in the appropriate **Title** column in the **Addenda Tables** dialog box. If you have edited the title of a nonactive file, the highlight bar returns to the active file.
5. Select **OK** to close the **Addenda Tables** dialog box. If necessary, select **OK** to close the **Calorimeter Files** dialog box.

6.7.5 Deleting an Addenda Table

You may delete any addenda table that is not the active addenda. Once you issue the **Delete** command, the addenda table is deleted permanently and may not be retrieved.

Complete the following steps to delete an addenda table:

1. Verify that the correct `.cal` file is active. Refer to Section 5.7.1. A `.cal` file contains only the addenda measurements taken for one puck.
2. Open the **Addenda Tables** dialog box by doing one of the following: (a) double-click on the **Addenda** status panel in the Heat Capacity control center, (b) select **Addenda Tables** in the **Files** tab in the Heat Capacity control center, or (c) select **Addenda** in the **Calorimeter Files** dialog box.
3. Click once on any row that is part of a nonactive addenda table. The row is highlighted, but the two arrows do not move to the left-most row column.
4. Select **Delete**, and then select **Yes**. The addenda table is deleted immediately, and the highlight bar returns to the active table.
5. Select **OK** to close the **Addenda Tables** dialog box. If necessary, select **OK** to close the **Calorimeter Files** dialog box.

6.7.6 Advance Addenda Table Manipulation

Under unusual circumstances it may be desirable to edit an addenda table. Clicking the **Addenda Entries** button in the **Addenda Tables** dialog will show the **Calibration Table Edit** dialog for the active addenda table. To change an entry, select the **Edit** tab, change an entry, or paste new entries in the text box following the existing format, then click the **Parse** button. When you are finished, click the **OK** button. To save the addenda, follow the instructions in section 5.7.4 to enable advanced table operations from the **Calorimeter Files** dialog. Then, select the **Advanced >> Save Active Calibration** command. You will also be prompted to save changes to the `.cal` file when you exit the software or select another `.cal` file.

Heat Capacity Measurement

7.1 Introduction

This chapter contains the following information:

- Section 7.2 presents an overview of sample heat capacity measurements.
- Section 7.3 explains how to measure sample heat capacity.
- Section 7.4 explains how to measure sample heat capacity in a magnetic field.
- Section 7.5 describes how to use the thermal cycle command.
- Section 7.6 summarizes and describes the heat capacity measurement parameters.
- Section 7.7 describes the heat capacity measurement process.

7.2 Overview of Heat Capacity Measurement

The Heat Capacity software application calculates the heat capacity of a sample by subtracting the addenda measurement from the total heat capacity measurement. The total heat capacity measurement is the measurement of the heat capacity of the sample, the grease, and the sample platform. The two measurements—one with and one without a sample on the sample platform—are necessary.

For accurate measurement of the sample heat capacity, you first apply a small amount of grease—just enough to hold the sample—to the sample platform, and then you measure the heat capacity of the grease and the platform: This is the addenda measurement, which is discussed in Chapter 6. Next, you mount the sample on the sample platform by pressing the sample onto the grease you have already applied to the platform. You then measure the heat capacity of the sample as described in this chapter. Automatic subtraction of the addenda, interpolated at each sample measurement temperature, is performed.

7.3 Measuring Heat Capacity

7.3.1 Measure the Addenda with Grease

Refer to Chapter 6 to measure the addenda. The addenda heat capacity measurement includes any grease you will use to hold the sample on the sample platform. Section 6.2 presents an overview of the addenda measurement. Section 6.3 explains how to measure the addenda.

If you do not require an accurate measurement of the sample heat capacity, or if you can precisely reproduce the amount of grease used from sample to sample, you may reuse the same addenda table for different samples, rather than remeasure the addenda for each sample. Section 6.7.1 explains how to select an addenda table.

If you are going to perform measurements in magnetic fields, you may need to measure the addenda for each of the fields of interest prior to mounting your sample. See Section 7.4 for more information on measuring heat capacity in a magnetic field.

7.3.2 Prepare the Sample for Measurement

1. Examine the sample to locate its flattest side, and then examine the flattest side of the sample. Look for slight surface irregularities or heavy oxidation, which impede thermal contact between the sample and sample platform.
2. Polish or sand the flattest side of the sample if you notice surface irregularities or oxidation. It is likely that the flattest and cleanest surface of the sample will achieve the best thermal contact with the sample platform.
3. Weigh the sample if you are measuring specific heat or Debye temperature. The software must know the mass of the sample in order to compute specific heat or Debye temperature. Section 1.4.1 discusses sample sizes that Quantum Design recommends you use with the Heat Capacity option.
4. Locate the puck's serial number, which is written on the green fiberglass connector that is at the base of the puck (see Figure 3-2). You need to know the serial number to select the `.cal` file that has been created for the puck.

7.3.3 Mount the Sample on the Sample Platform

1. Plug in the small vacuum pump that is supplied with the sample-mounting station. If the pump has a power switch, turn it on. Otherwise the pump will turn on when you plug it in. Verify that the sample-mounting station is receiving vacuum.
2. Slide the thermal radiation shield off the puck if the shield covers the puck.
3. Place the puck, with the sample platform facing upward, inside the sample-mounting station's puck holder. Rotate the puck until the index key slips into the notch at the rear of the holder, and then *gently* push the puck into the holder. Verify that the sample platform is properly seated on the platform holder.
4. Pivot the puck interlock arm toward the puck so that it grabs and immobilizes the puck. When the interlock arm is against the puck, vacuum pulls the sample platform downward.

A hissing sound in the puck holder indicates a poor seal between the sample platform and holder. To eliminate the leak, remove the puck from the holder, clean off all debris that is under the puck, and then place the puck back inside the holder.

5. Position the sample-mounting station and the puck below a wide field stereo microscope and a strong light, if desired.

CAUTION!

Caution and a steady hand are required to mount and remove samples. You risk breaking the puck wires unless you work very carefully *and* you feel comfortable using tweezers to manipulate samples under the microscope. If you break a wire, you must replace the entire puck frame and platform along with the wires. Section 10.4.1 explains this procedure.

6. Use tweezers to place the sample, with its flattest side downward, on top of the grease that is on the sample platform. Use the blunt edge of a cotton-tipped applicator to *gently* push the sample downward if the sample does not rest flat on the platform. The best thermal contact to the sample occurs when a small amount of grease is squeezed out around all sides of the sample. If you do not see this grease, the surface of the sample is probably not flat or it contains a burr.
7. Push the puck interlock arm away from the puck.
8. Pull the puck off the puck holder. To verify that the sample is attached to the sample platform, you may turn the puck upside down. Be prepared to catch the sample if it is not securely attached to the platform. If the sample falls off, return to step 3.
9. Hold the puck so that the sample platform is facing upward. Slide the thermal radiation shield over the top of the puck, and then twist the shield to verify that it is securely in position. See warning in Section 11.3.6.

Note: Use the puck adjustment tool (see Section 9.2) on the puck after the puck has been inserted in the sample chamber approximately 10 times. By adjusting the tension in the chuck fingers, the puck adjustment tool ensures solid thermal contact between the puck and the heater block located at the bottom of the sample chamber.

10. Use the broken edge of a cotton-tipped applicator to apply a small amount of Apiezon H Grease to each chuck finger (Figure 3-1) if the fingers are dry. Apply only enough H Grease to make the fingers slightly sticky.
11. Unplug the vacuum pump if you will not immediately reuse the sample-mounting station.

7.3.4 Insert the Puck

1. Select the **Installation Wizards** tab in the Heat Capacity control center.
2. Select **Prepare Sample Measurement**. The **Installing Puck for Sample Measurement** dialog box opens.
3. Select **Open Chamber** to vent the sample chamber and warm it to room temperature.
4. Insert the puck into the sample chamber when the on-screen instructions prompt you to do so. The *Physical Property Measurement System: Hardware Manual* discusses puck insertion in detail.

5. Install the baffle assembly. Verify that the charcoal holder is attached to the bottom of the assembly. Refer to Figures 3-7 and 3-8B.

CAUTION!

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

6. Insert the baffle assembly into the sample chamber.
7. Select **Purge** to purge and seal the sample chamber.
8. Select **Next**. Then enter the puck's serial number.
9. Select **Next**. The **Current Calibration File** panel opens.

7.3.5 Select a Calibration File

Review the information identifying the active `.cal` file in the **Current Calibration File** panel (see Figure 7-1). The panel displays summary information—serial number, creation date, name, title, temperature range, and the number of associated addenda tables—for the active `.cal` file. The serial number identified in the **Current Calibration File** panel should match the serial number you entered previously. A warning message appears at the bottom of the panel if the two numbers do not match.

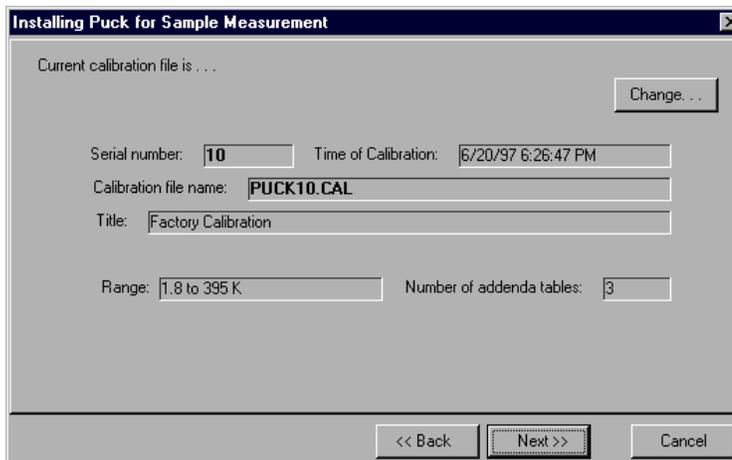


Figure 7-1. **Current Calibration File** Panel in Puck Installation Wizard

If the serial numbers match, indicating that the correct `.cal` file is selected, select **Next** to open the **Current Addenda Table** panel.

If the serial numbers do *not* match, indicating that the correct `.cal` file is not selected, you must select the correct `.cal` file. Do the following:

1. Select **Change** in the **Current Calibration File** panel. The **Calorimeter Files** dialog box opens (see Figure 5-1 or 5-7).

2. Locate the `.cal` file that includes the puck's serial number. The serial number appears in the name of the file and in the **Ser#** column. If necessary, adjust the size of the columns or use the scroll bar to view all information in the columns.
3. Select the correct `.cal` file as follows: (a) click anywhere on the row that summarizes the data saved in the correct `.cal` file, (b) click on **Select**, and (c) click on **OK** in the pop-up message. The file is selected only when the row is highlighted and the two arrows appear to the left of it.
4. Select **OK** in the **Calorimeter Files** dialog box. The **Current Calibration File** panel now displays all data stored in the `.cal` file you have activated. Any warning message disappears if the serial numbers match.
5. Select **Next** to open the **Current Addenda Table** panel.

7.3.6 Select an Addenda Table

Review the information identifying the active addenda table in the **Current Addenda Table** panel (see Figure 7-2). The panel displays summary information—table ID number, creation date, measurement parameters, title, temperature range, magnetic field at which the measurement was obtained, and any additional offsets to be applied—for the active addenda table. A warning message appears at the bottom of the panel if the active table contains no measurement data.

Although you normally want to select an addenda table at this point, it is also possible to select an addenda table from a sequence by executing the **Switch Addenda** sequence command. Refer to Section 6.7.2 for more information on changing addenda tables from a sequence.

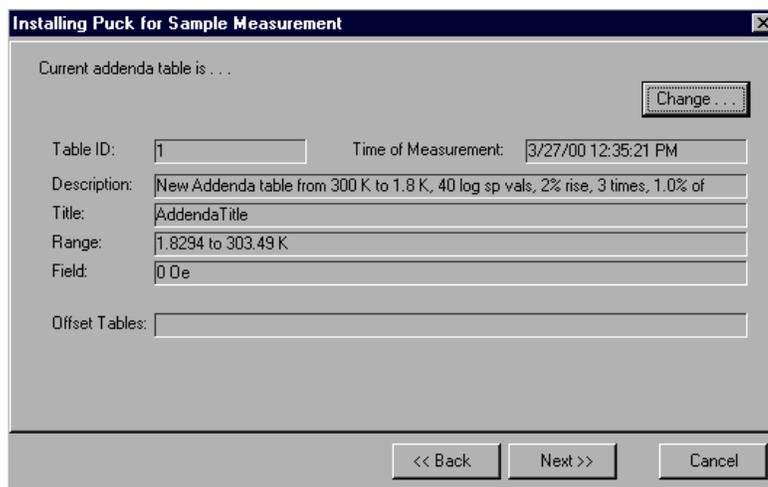


Figure 7-2. **Current Addenda Table** Panel in Puck Installation Wizard

If you want to use the active addenda table, select **Next** to open the **Puck Test Results** panel.

If you want to activate another addenda table, do the following:

1. Select **Change** in the **Current Addenda Table** panel. The **Addenda Tables** dialog box opens (Figure 6-9).
2. Select an addenda table as follows: (a) click anywhere on the row that summarizes the data saved in the addenda table, (b) click on **Select**, and (c) click on **OK** in the pop-up message. The table is selected only when the row is highlighted and the two arrows appear to the left of it.

3. Select **OK** in the **Addenda Tables** dialog box. The **Current Addenda Table** panel now displays all data stored in the addenda table you have activated.
4. Select **Next** to open the **Puck Test Results** panel.

7.3.7 Test the Puck

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

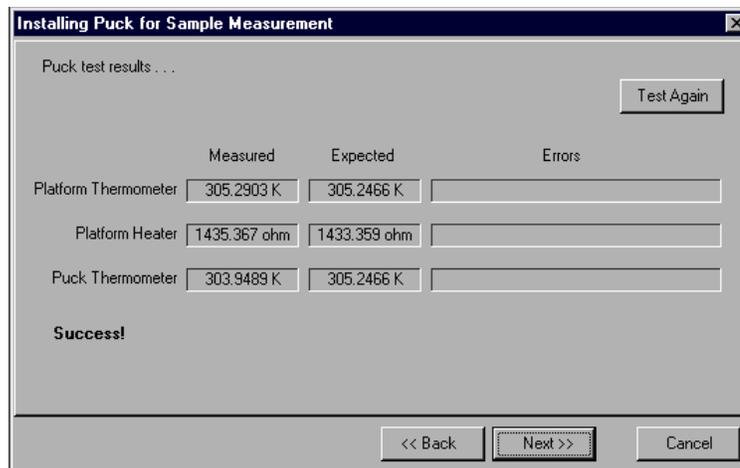


Figure 7-3. **Puck Test Results** panel in puck installation wizard. The displayed values are in kelvin for the two thermometers and in ohms for the heater.

A failure might indicate a broken puck wire or a loose or unplugged cable or that the temperature of the puck is changing too rapidly. If a puck wire is broken, you must replace the puck frame. Refer to Section 9.4.1.

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

7.3.8 Select a Data File

Note: In certain cases, it might be useful to append new measurements to the end of an existing file. When you append to an existing file, the original header information and the measurement units that were originally selected are extracted from the header of the file. You are not prompted to enter this information when you append to an existing data file.

Review the **Header Information** in the **Data File Name** panel. The **Header Information** lists the information stored in the header of the active data file.

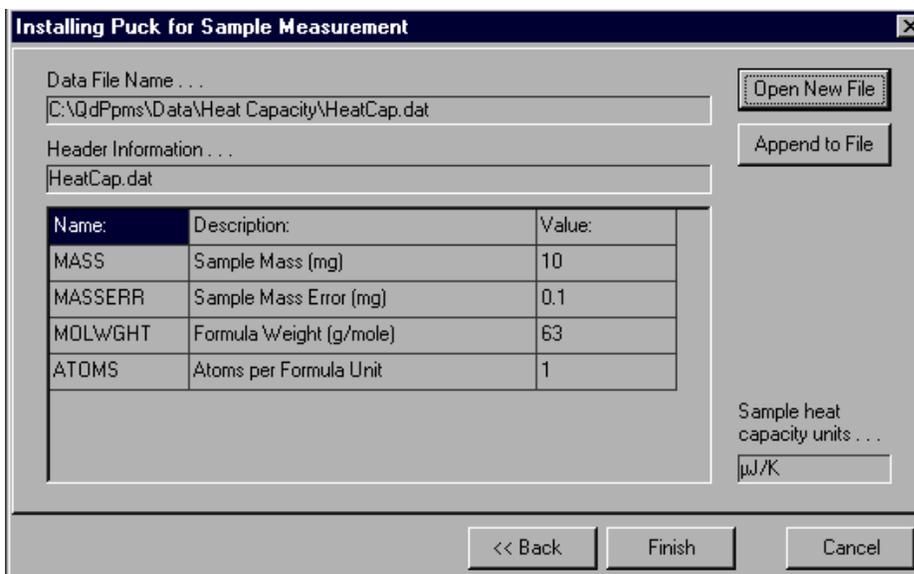


Figure 7-4. **Data File Name** panel in puck installation wizard. The name and location of the active data file are identified at the top of the panel. The information in the header of the active file is displayed below the **Header Information** box. If no data file is active, the **Data File Name** and **Header Information** boxes are blank.

- If you want to use the data file that is currently active, select **Finish**. The **Data File Name** panel closes.
- If you want to append data to a different data file, select **Append to File**, select the file, and then select **Open**. The **Header Information** in the **Data File Name** panel lists the information stored in the header of the file you have just selected. Select **Finish** to close the **Data File Name** panel.
- If you want to create a new data file, do the following:

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

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

3. Enter a different title for the graph view of the file, if necessary. By default, the file name is also the title of the graph view.
4. Review the **Sample Information** that will be saved to the header of the file. Define the **Sample Information** as necessary. Leaving the **Sample Information** fields blank is permissible, but allows the resulting heat capacity measurements to be expressed in only heat capacity units rather than specific heat units. Providing more information increases the choices of specific heat units for expressing the sample data. The mass error is used in calculating the specific heat error during a measurement.

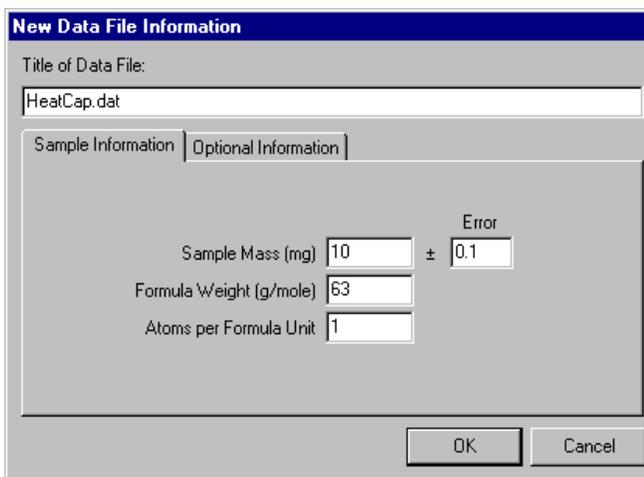


Figure 7-5. Sample information saved to data file header. In this example, all sample information is defined.

5. Review the user-defined **Optional Information** that will be saved to the file header. Select **Configure List** to add, edit, or delete optional items. See Section 4.7.2.1 for more information.
6. Select **OK**. The **Units** dialog box opens if you defined **Sample Information** to be saved in the file header. If you did not define **Sample Information**, the **Data File Name** panel opens again.

7. If the **Units** dialog box is open, select the measurement units, and then select **OK**.

The **Data File Name** panel opens again. The **Header Information** now displays the header of the data file you have just opened. The measurement units you have selected appear in the lower right corner.

8. Select **Finish** to close the **Data File Name** panel.

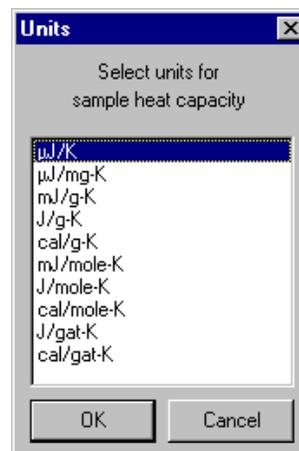


Figure 7-6. Units available for expressing sample data. The number of user-defined sample information fields determines which units are available. In this example, all possible unit selections are available, because all sample information was defined.

7.3.9 Run the Heat Capacity Measurement

1. Select the **Measurement** tab in the Heat Capacity control center.

2. Select **Measure Sample Heat Capacity vs. Temperature**. The **Heat Capacity Versus Temperature** dialog box opens. You use this dialog box to specify the parameters for the sample heat capacity measurement. The dialog box displays the values used for the last sample heat capacity measurement. The parameters are organized into a **Setup** tab (Figure 7-7) and an **Advanced** tab (Figure 7-8). Section **Error! Reference source not found.** explains the parameters in more detail.

CAUTION!

Under certain circumstances, residual helium may be absorbed by components of the calorimeter if the calorimeter is left below 6 K for several hours prior to a measurement. If this is the case, it is recommended that the system be thermally cycled to above 6 K prior to a measurement or that you measure with decreasing temperatures starting above 6 K.

3. Select the **Setup** tab. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

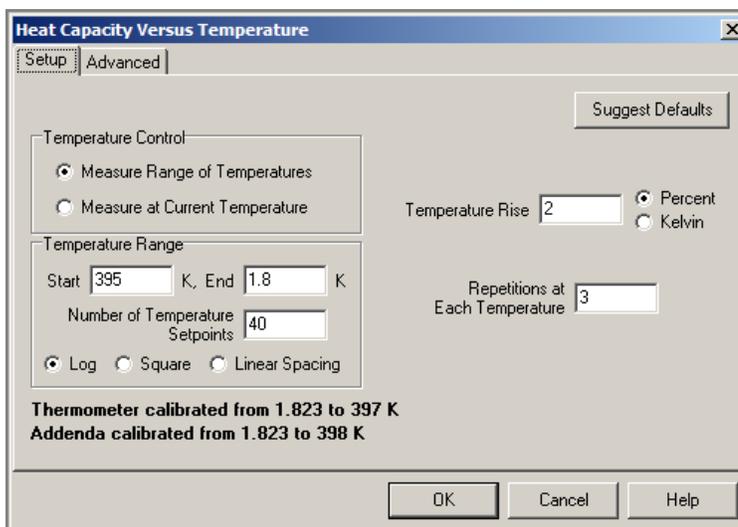


Figure 7-7. **Setup** Tab in **Heat Capacity Versus Temperature** Dialog Box

4. Enter the starting value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range, which are displayed in the lower left corner of the dialog box. The first measurement is taken at the starting temperature value.
5. Enter the ending value of the temperature range. Select a value that is within the thermometer calibration range and the addenda temperature range. The last measurement is taken at the ending temperature value.
6. Enter the number of temperature values, including the starting and ending temperature values, for which you want to take measurements.

Note: Selecting exactly matching measurement points for the sample heat capacity measurement and the addenda measurement is unnecessary. During the sample heat capacity measurement, the software uses polynomial interpolation of the active addenda table at the measurement temperatures for determining the appropriate addenda to subtract.

7. Select the type of spacing that is used to separate the temperature values.
8. Enter a value for the temperature rise.
9. Enter the number of times a measurement is repeated at each temperature.
10. Select the **Advanced** tab if you want to review the advanced measurement parameters. Quantum Design recommends that only default advanced measurement parameters be used. Selecting the **Suggest Defaults** button in the tab inserts suggested values for the measurement parameters.

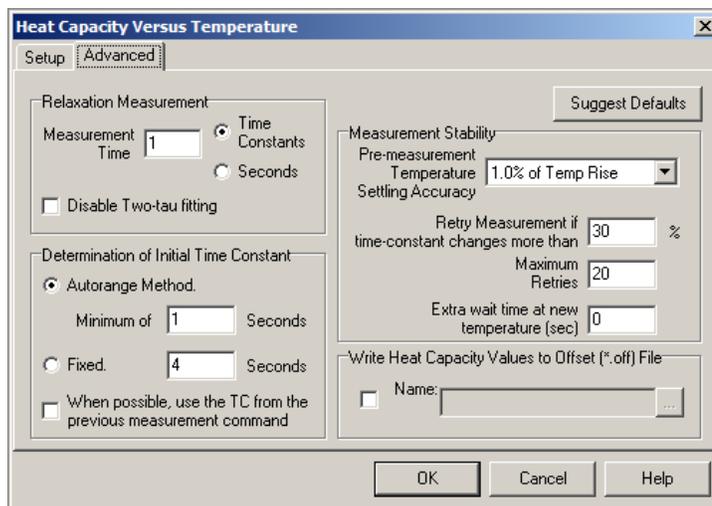


Figure 7-8. **Advanced** Tab in **Heat Capacity Versus Temperature** Dialog Box

11. Select the **OK** button at the bottom of the **Heat Capacity Versus Temperature** dialog box. The sample heat capacity measurement begins if the temperature range you defined is within the thermometer and addenda calibration temperature range.

As soon as the measurement begins to run, the Measurement Status Viewer opens to show the measurement's progress. The name of each task that is part of the measurement appears, as it is performed, in the message list box at the bottom of the Viewer. When the software performs a new measurement, it presents the data in the measurement-field panels and plots the data as a graph. Section 4.5 discusses the Measurement Status Viewer in detail.

The bottom status panel in the Heat Capacity control center also indicates the name of each task as the task is performed.

Section 7.7 describes the heat capacity measurement process in more detail.

12. Wait for the measurement to finish. A measurement lasts several hours or even days. When the measurement is complete, the **Idle** and **Done** messages appear in the bottom status panels in the Heat Capacity control center.

7.3.9.1 PAUSING OR ABORTING A MEASUREMENT

You may pause and resume a sample heat capacity measurement or abort a measurement at any time.

To pause a measurement, select **Pause** in the Measurement Status Viewer.

To abort a measurement, select **Abort** in the Measurement Status Viewer. You may abort a paused measurement.

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

7.3.10 Remove the Puck

Refer to the *Physical Property Measurement System: Hardware Manual* to remove the puck from the sample chamber.

7.3.11 Remove the Sample from the Sample Platform

1. Plug in the small vacuum pump that is supplied with the sample-mounting station. Verify that the sample-mounting station is receiving vacuum.
2. Slide the thermal radiation shield off the puck if the shield covers the puck.
3. Place the puck, with the sample platform facing upward, inside the sample-mounting station's puck holder. Rotate the puck until the index key slips into the notch at the rear of the holder, and then *gently* push the puck into the holder. Verify that the sample platform is properly seated on the platform holder.
4. Pivot the puck interlock arm toward the puck so that it grabs and immobilizes the puck. When the interlock arm is against the puck, vacuum pulls the sample platform downward.
A hissing sound in the puck holder indicates a poor seal between the sample platform and holder. To eliminate the leak, remove the puck from the holder, clean off all debris that is under the puck, and then place the puck back inside the holder.
5. Position the sample-mounting station and the puck below a wide field stereo microscope and a strong light, if desired.

CAUTION!

Caution and a steady hand are required to mount and remove samples. You risk breaking the puck wires unless you work very carefully *and* you feel comfortable using tweezers to manipulate samples under the microscope. If you break a wire, you must replace the entire puck frame and platform along with the wires. Section 10.4.1 explains this procedure.

6. Use tweezers to gently slide the sample off the platform. Never pull the sample directly up off the platform. If you pull the sample directly upward, you may pull the platform off its mount and break the wires.
7. Push the puck interlock arm away from the puck.
8. Pull the puck off the puck holder if you will not immediately mount another sample onto the puck.
9. Unplug the vacuum pump if you will not immediately reuse the sample-mounting station.

7.4 Measurements in a Magnetic Field

Heat capacity can be measured in a magnetic field. However, there are two effects that must be considered before performing such a measurement. First, the sample platform thermometer exhibits a magneto-resistance effect that causes the resistance of the thermometer to change with magnetic field in addition to the normal temperature dependence. The resulting temperature error must be corrected using the calibration procedure described in Chapter 5. In addition, you must be careful to properly account for the magnetic field dependence of the addenda heat capacity. The heat capacity of the platform can depend on the applied field. Hence, before measuring sample heat capacity in a magnetic field, it may be necessary to measure the addenda at each of the desired fields and then select the appropriate addenda, depending on the magnetic field, for the sample measurement. See section 6.2.5 for more information. This procedure should not be necessary for VersaLab systems.

If you are using the Helium-3 insert for performing heat capacity measurements, you may also need to prevent the heat capacity platform from oscillating in the magnetic field during measurements. Due to the orientation of the heat capacity platform on the Helium-3 insert, very small vibrations in the platform may cause large amounts of noise in the measurement. See Chapter 8 for more information on using the Helium-3 insert for measuring heat capacity.

To run a heat capacity measurement in a magnetic field, you complete the following basic steps:

1. Assuming the puck is already calibrated for zero-field operation, perform a secondary field calibration on the same puck at the specific fields at which you will be performing measurements. Refer to Section 5.4. This portion of the calibration should only be necessary from about 20 K to the lowest temperature. Also, it should only be required once for a specific puck.
2. Once the puck is calibrated, prepare for an addenda measurement by applying grease to the platform in the usual way. Then perform an addenda measurement at each of the desired fields as described in Section 6.3. This may not be necessary depending on temperature range, puck type, and accuracy requirements (see Section 6.2.5).
3. Mount your sample and measure the sample heat capacity. At each field, set the field to the desired value. Then select the corresponding addenda table as created above. Finally, measure the sample heat capacity. The **Switch Addenda** command as described in Section 6.7.2 can be used to automatically select the addenda under sequence control.

7.4.1 Example: Measuring Sample Heat Capacity at 0 Oe and 90,000 Oe

It is assumed that the puck has already been calibrated for magneto-resistance at 90,000 Oe.

1. Prepare the addenda by applying a small amount of grease as usual.
2. From the **Installation Wizards** tab press the **Prepare Addenda Measurement** button and follow the instructions to install the calorimeter into the sample chamber, select the appropriate calibration file, test the puck, and specify an optional output file.
3. Prepare a sequence that will perform the following functions:
 - **Set Field** to 90000 Oe
 - **Wait** for Field
 - **New Addenda** from 100 K to 0.5 K
 - **Set Field** to 0 Oe

- **Wait** for Field
 - **New Addenda** from 100 K to 0.5 K
4. Execute the sequence. Note that each addenda command generates another addenda table. Hence after execution is complete, there will be two new addenda tables. The current one will correspond to zero magnetic field since it was the last one acquired. The one at 90000 Oe will be the next one in the list of addenda tables.
 5. From the **Installation Wizards** tab press the **Prepare Sample Measurement** button and follow the instructions to first remove the calorimeter from the sample chamber, mount the sample, install the calorimeter into the sample chamber, select the appropriate calibration file, test the puck, and specify an optional output file.
 6. Once the installation is complete, prepare a sequence that will perform the following functions:
 - **Sample HC** from 100 K to 0.5 K
 - **Set Field** to 90000 Oe
 - **Wait** for Field
 - **Switch Addenda** to the 90000-Oe addenda table
 - **Sample HC** from 100 K to 0.5 K
 7. Execute the sequence. Note that the first command assumes that the zero-field addenda is already selected and that the field is still at zero.

7.5 Thermal Cycle Temperature Command

When measuring heat capacity at low temperatures, it is sometimes useful to warm the sample above a certain critical temperature and cooling it again before performing a heat capacity measurement. For example, for superconducting samples, it might be useful to warm the sample above its critical temperature after changing the magnetic field. Another time when this might be useful is for burning off helium films from porous sample at low temperatures (see Section 11.3.1).

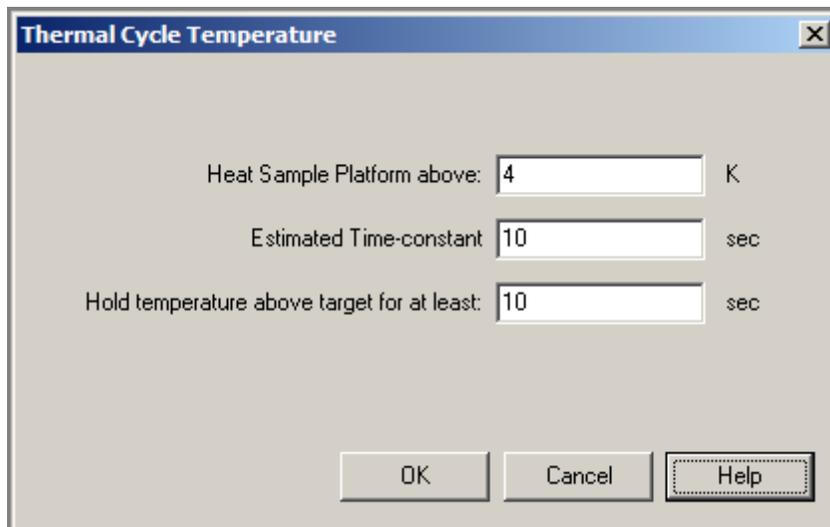


Figure 7-9 Thermal Cycle Temperature Dialog

A sequence command called Thermal Cycle Temperature (Figure 7-9) is provided for warming the platform temperature up to a predetermined temperature and holding it at that temperature for a designated amount of time. While it is possible to cycle the sample temperature using standard temperature control commands, this command is significantly faster since it only warms the sample and sample platform instead of the calorimeter puck and sample chamber. It is particularly convenient when used with the DR insert (Chapter 9) because of the relatively longer time required to change temperatures of the insert. The Thermal Cycle Temperature sequence command also requires that you specify an estimated time constant for more rapid convergence to a temperature that exceeds the target without warming the sample any more than necessary. The maximum temperature that can be reached will vary with temperature and calorimeter, but will always be greater than 20 K.

7.6 Summary of Heat Capacity Parameters

Table 7-1. Parameters in **Setup** Tab in **Heat Capacity Versus Temperature** Dialog Box

PARAMETER	DEFINITION
Temperature Control	How measurement temperature is determined. Select Measure Range of Temperatures to specify a starting and ending temperature. Select Measure at Current Temperature to perform the measurement while stable at the current temperature setpoint. This can be useful for sequence programming where the measurement command appears in a Scan Temperature or Scan Field loop.
Temperature Range Starting Value	Starting value for temperature range. First measurement is taken at this temperature. Heat capacity temperature range should be within temperature range of both thermometer calibration and addenda.
Temperature Range Ending Value	Ending value for temperature range. Last measurement is taken at this temperature. Heat capacity temperature range should be within temperature range of both thermometer calibration and addenda.
Number of Temperature Values	Number of temperature values, including starting and ending value, at which measurements are taken.
Spacing for Temperature Values	Logarithmic, square, or linear spacing between temperature values. Logarithmic spacing equally spaces logarithms of temperature values. Square spacing equally spaces squares of temperature values. Linear spacing equally spaces temperature values.
Temperature Rise	Temperature rise produced by heater-on cycle of heat capacity measurement. Large temperature rise reduces scatter. Small temperature rise is necessary if features such as peaks must be resolved. If temperature rise is too large, it may exceed current limitation of heater driver, or it may produce inaccurate measurement if heat capacity varies substantially over the range produced by the rise. Typical value for temperature rise is 2%. Temperature rise is either given in kelvin or as a percentage of sample temperature at each measurement, depending on user selection. The application automatically determines the heater current needed to produce this temperature rise using the wire conductance tables in the .cal file.
Number of Repetitions Per Measurement	Number of times a measurement is repeated at each temperature. A value of two or more is recommended since this gives a good indication of the self-consistency of measurements.

Table 7-2. Parameters in **Advanced** Tab in **Heat Capacity Versus Temperature** Dialog Box

PARAMETER	DEFINITION
<p>Relaxation Measurement: Measurement Time</p>	<p>Amount of time, including heater-on and heater-off sequences, used for a single relaxation measurement. Measurement time is specified in time constants or seconds. When specified in time constants (the default) the measurement time window is properly scaled for possibly vastly different magnitudes of heat capacity as the temperature changes. When specified in seconds, the measurement time is fixed and does not vary. This setting may help reduce measurement scatter under some circumstances where internal time-constants in the sample cause the reported heat capacity to vary with measurement time.</p> <p>If sample is thermally well attached to the platform, a smaller value for measurement time may reduce scatter. If poorly attached, longer times may be indicated.</p> <p>A typical value for measurement time is 1 time constant. If the puck type is "Standard," the recommended value is 2 time constants for very small samples. This is because the standard puck always uses a measurement time of 2 time constants for addenda measurements.</p>
<p>Relaxation Measurement: Disable Two-tau fitting</p>	<p>When checked, this setting forces the measurement to be fit using the "Simple Model" (single time constant) as described in Section 4.3.3. Under some circumstances, the scatter in the measurement results may be lowered with this setting. Normally, the Two-tau model is used.</p>
<p>Relaxation Measurement: Allow TCs less than Addenda TCs</p>	<p>When checked, this setting allows time – constants for a sample measurement that are less than those for the addenda. Normally, this would be an error condition indicating extremely poor thermal contact between the sample and platform and the system discards such measurements. However, in unusual cases, say when the addenda table has values that make the reported sample heat capacity negative, checking this box will allow the system to report such values.</p>
<p>Determination of Initial Time Constant: Autorange/Fixed Method</p>	<p>This setting governs the determination of the first time constant when the Measurement Time is given in time constants. It only affects the first measurement when a new Heat Capacity Versus Temperature command is executed. When Autorange Method is selected, a trial heat pulse is used. The Minimum of setting makes sure the first measurement is not too short. The Fixed setting is will use the given time as the initial guess. This can save time when you know, within a factor of ten or so, the actual time constant.</p>
<p>Determination of Initial time Constant: When possible, use the TC from the previous measurement command</p>	<p>When checked, this setting will make the initial time constant for this measurement the same as the last measured time constant from previous Heat Capacity Versus Temperature command. This is particularly useful in conjunction with the Measure at Current Temperature setting when the measurement command is part of a Scan Temperature or Scan Field sequence loop. If this is the first measurement since the Heat Capacity option software was activated, the Autorange/Fixed settings are used.</p>
<p>Measurement Stability: Settling Accuracy</p>	<p>Determines how long software waits for temperature stability before taking each measurement. Settling accuracy is given as a percentage of temperature rise. If sample-platform temperature changes between successive readings by less than the settling-accuracy value, temperature is stable enough to proceed with measurement. Small settling accuracies lead to better temperature reproducibility at a specific temperature, though not necessarily better heat capacity values. That is, the temperature scatter will be smaller when this value is smaller but the heat capacity values will still fall on the correct curve. However, the measurement will take longer with a smaller value. A typical value for settling accuracy is 1%.</p>

Measurement Stability: Retry Percentage	Determines how much the time constant is allowed to change from measurement to measurement. For example, a typical value of 30% would mean that if previous relaxation measurement had a time constant of 1 second, and the current measurement was greater than 1.3 or less than 0.7, the current measurement would be discarded and the measurement repeated with the latest acquired time constant. A smaller value may reduce temperature hysteresis effects with some samples by requiring better self-consistency between the assumed time constant used for the "Measurement Time" and the actual time constant determined from the fit.
Measurement Stability: Maximum Retries	Maximum number of times the above condition will discard a measurement before doubling user-specified "Measurement Time." This is an attempt to recover self-consistency. The assumption is that sample is possibly very poorly attached or that specified Measurement Time is much less than 1 and is too short to discern a good value for the time constant when the data is fit. All subsequent measurements will be performed with the longer time constant.
Measurement Stability: Extra wait time at new temperature (sec)	After the PPMS declares stability at a new temperature, the software will wait for the indicated time before starting the measurement. Subsequent measurements at the same temperature do not wait.
Write Heat Capacity Values to Offset File	If this is selected, the heat capacity values obtained from this procedure will be written to the specified <code>.off</code> file in addition to the usual output data file. The offset files reside in the <code>TempCal</code> directory and are used in subsequent measurements as an additional contribution to the addenda. See Section 6.7.3 for more on using these offset tables.

7.7 Description of Heat Capacity Measurement Process

When a sample heat capacity measurement begins, the turbo pump evacuates the sample chamber, and then the chamber temperature stabilizes at the user-defined starting temperature value.

Before the Heat Capacity software can perform the first heat capacity measurement, it must obtain an estimate of the first characteristic heating and cooling time, or *time constant*, of the sample platform. The method used depends on the setting of the **Determination of Initial Time Constant** parameter as described in table 7-2. The **Measurement Time** parameter is expressed in units of time constants, so the software must know the time constant to determine the data acquisition time of the first measurement. Depending on the sample's size and temperature, the time constant could be as small as 100 milliseconds or greater than 100 seconds.

If the **Autorange Method** is chosen, the software reads the active `.cal` file, and by using the value of the thermal conductance of the puck wires, adjusts the temperature of the platform heater in order to create the requested temperature rise. The Heat Capacity Controller uses a timer to monitor the temperature rise until the rise reaches approximately half of its eventual value. The elapsed time that the controller monitors is the initial estimate of the time constant. After the software takes one heat capacity measurement, it estimates the time constant from the measurements. The software usually estimates the initial time constant only once for a given temperature sweep.

Using the estimate of the time constant, the software waits for the platform temperature to stabilize before it takes each measurement. The software monitors the drift rate, which is the slope of temperature versus time, of the platform thermometer at single time constant intervals until it detects no change or detects a change that is less than the change specified by the **Settling Accuracy**

parameter. Once the drift rate is stable, the software applies the heater pulse and records the temperature and heater power, as a function of time, into the Heat Capacity Controller buffer.

The software uses several parameters to fit the time trace of the thermometer response to the two-tau model (see Section 4.3.1.2). This includes an interpolated value for the addenda from the previous addenda measurement.

Before the software takes the next measurement, it appends, as a single record, the relevant data from the fit to the open data file. The software writes the raw data from the measurement and the fit to temporary files in the `Heat Capacity\LogFiles` directory. Section 4.7.4 discusses `.raw` files. Section 4.7.5 discusses `.fit` files.

Operation with the Helium-3 System

8.1 Introduction

This chapter contains the following information:

- Section 8.2 discusses considerations to keep in mind when performing Heat Capacity measurements with the Helium-3 system.
- Section 8.3 explains how to measure the addenda when the Helium-3 insert is installed.
- Section 8.4 explains how to measure the sample heat capacity when the Helium-3 insert is installed.
- Section 8.5 explains how to calibrate the Helium-3 calorimeter puck.

8.2 Special Considerations for Performing Heat Capacity Measurements with the Helium-3 Insert

The PPMS Helium-3 Refrigerator System option (Model P825) and the Heat Capacity option were designed to function together. Performing heat capacity measurements with the Helium-3 system involves the same setup as used without the Helium-3 insert. You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before you proceed with a corresponding measurement using the Helium-3 insert. As with standard pucks, before you measure the heat capacity of a sample, you must first measure the addenda to create a table. Then you can mount the sample on the puck and perform the measurement.

This section describes some of the key differences between heat capacity measurements that are performed with and without the Helium-3 insert. For detailed instructions on calibrating a calorimeter puck for use with the Helium-3 system, refer to Section 8.5.

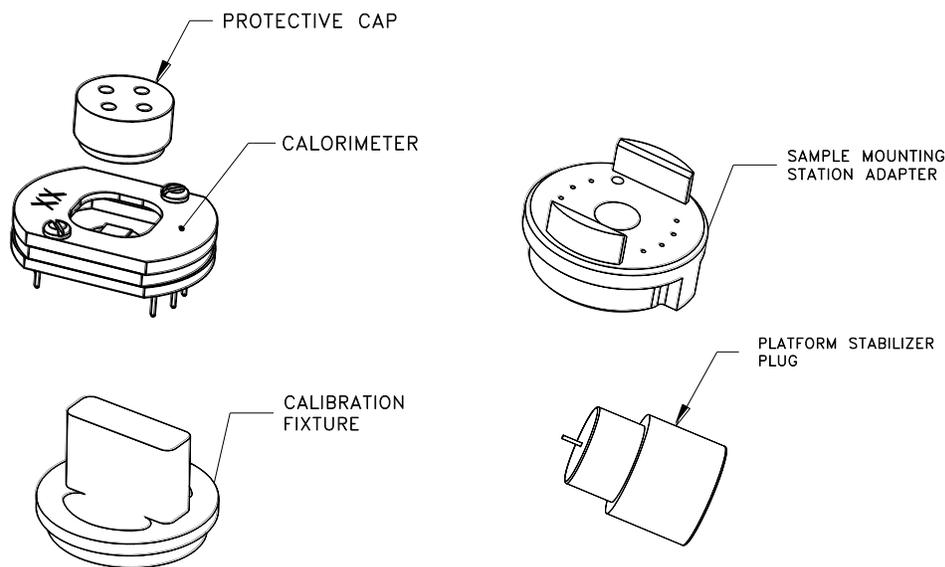


Figure 8-1. Heat Capacity Sample Mount, Mounting Adapter, Calibration Fixture, and Platform Stabilizer

8.2.1 Helium-3 Calorimeter Puck

The calorimeter puck (Figure 8-1) used on the Helium-3 insert is based on the frame assembly used with the standard Heat Capacity puck. The thermometer used on the Heat Capacity platform is specially designed to function at temperatures down to 0.35 K. This thermometer also limits the maximum temperature to 350 K.

Also, because of the difficulty of achieving good thermal contact between materials at these temperatures, the contact wires to the platform have been designed to provide less thermal anchoring to the surroundings. Instead of eight wires for suspending the sample platform, only four wires are used. Furthermore, these wires have approximately half the cross-sectional area of the wires in the standard pucks. This effectively slows down the measurement and thus provides better thermalization of the sample. Unfortunately, this also makes these platforms very delicate. Even slight bumps to the probe when the Heat Capacity puck is mounted can lead to a broken wire.

Due to the construction of the Helium-3 insert, mounting of the Heat Capacity puck differs according to orientation of the sample stage: the puck is mounted vertically in the PPMS for a Helium-3 insert with a vertical sample stage and upside-down for a Helium-3 insert with a horizontal sample stage. Because a mounted sample could slip off the platform in these orientations, a protective cap is provided that twists onto the puck (Figure 8-1). This cap prevents the sample from becoming lost in the unlikely event that the sample slips off. The sample is much more likely to slip off if you are using Apiezon N Grease at temperatures above 300 K. It is recommended that Apiezon H Grease be used at higher temperatures. The cap also serves as protection against putting a finger through the window during mounting and handling.

Another more obvious consequence of the vertical platform orientation is the relationship to the magnetic field. Refer to Section 8.2.2 below.

8.2.2 Measurements in Magnetic Fields

As described in Chapters 5, 6, and 7, the thermometer used on the platform produces temperature errors of up to 50% in magnetic fields of several tesla when used at the lowest temperatures. This magneto-resistance effect requires special handling to perform successful measurements in fields. The Helium-3 system thermometer has a correction built in for correcting temperature errors in the Helium-3 temperature control due to magnetic field. To obtain this correction for heat capacity measurements it is necessary to add magnetic field calibration data to the `.cal` file as described in section 5.4. Also, as described in Chapter 6, the heat capacity of the addenda changes with applied magnetic fields below about 10 K. Successful measurements in magnetic fields below about 2 K will require that an addenda table is measured for each of the corresponding magnetic fields of interest.

8.2.3 Vibration of the Sample Platform

8.2.3.1 VERTICAL SAMPLE-STAGE SYSTEMS

For Helium-3 systems with a vertical sample stage, the electronic measurement of the heat capacity platform thermometer is particularly sensitive to vibrations when a magnetic field is applied. Even the smallest vibrations can produce large disturbances in the measurement data. Such vibrations will appear as an oscillating temperature superimposed on the platform temperature response. The magnitude of this oscillation varies from calorimeter to calorimeter and hence may not be a problem with some pucks.

For this reason, the platform stabilizer (Figures 8-1 and 8-2) has been designed to touch the back of the heat capacity platform with a nylon filament to prevent oscillations without excessive thermal contact. For best results, use it for all heat capacity measurements.

Complete the following steps to mount the platform stabilizer:

1. Mount your sample or prepare your addenda in the usual way and mount the puck onto the Helium-3 insert as described below.
2. If the cap on the puck does not have vent holes incorporated into it, remove the cap from the puck after it is installed. Since the stabilizer plug will block the other hole, the cap is removed so the gas can be evacuated effectively.
3. Remove the protective vinyl cover from the platform stabilizer plug and apply H Grease at the shoulder of the plug as shown in Figure 8-2 on the following page. This grease is used to hold the stabilizer plug in the sample mount.
4. Carefully insert the platform stabilizer into the hole in the backside of the Helium-3 sample stage so that the nylon filament touches the backside of the heat capacity platform. Verify that there is enough grease to hold the plug so that the probe can be inserted without the plug falling out. Only push in the plug far enough to make contact with the platform.

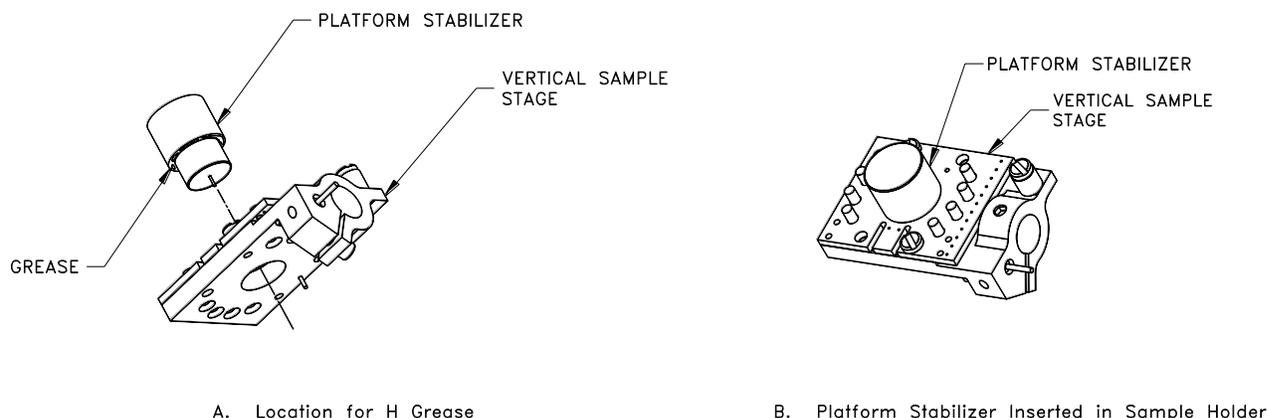


Figure 8-2. Applying H grease to the platform stabilizer and inserting the platform stabilizer into the vertical sample stage.

8.2.3.2 HORIZONTAL SAMPLE-STAGE SYSTEMS

For Helium-3 systems with a horizontal sample stage, vibration in a magnetic field usually does not produce a significant problem. Therefore, the platform stabilizer is not used with Helium-3 systems that have horizontal sample stages. If you experience vibrations with a horizontal platform, it may not affect the resulting measurement data. If it is a problem, please contact Quantum Design.

CAUTION!

The platform stabilizer is not compatible with Helium-3 systems that are equipped with the horizontal sample stage. Attempting to use the platform stabilizer with such systems could result in damage to a heat capacity puck.

8.2.4 Using the Sample-Mounting Station

When you apply grease to the sample platform when you are measuring the addenda or when you are mounting samples, it is necessary to use the sample-mounting station to immobilize the platform. This is particularly critical because of the fragile wires used for support.

A special adapter (part number 4092-625) is provided for the purpose of allowing you to use the standard sample-mounting station with the Helium-3 calorimeter pucks. The following steps describe how you use the sample-mounting station:

1. Remove the calorimeter puck from the Helium-3 insert if the puck is still on the insert.
2. Attach the calorimeter puck to the sample-mounting station adapter (see Figure 8-1). It is a good idea to keep the protective cap on during this step. The eight pins should slip easily into the eight holes and the puck should fit nicely between the two raised sections of the adapter.
3. Use the two long screws through the back side of the adapter to snugly hold the calorimeter onto the adapter. For convenience, coat the screws with H grease before you insert them into the holes in the adapter. The grease keeps the screws from falling out when the puck is not mounted.

Once the two parts are held snugly together, you can start the pump for the sample mounting, insert the assembly onto the mounting station, and completely close the lever arm as you would with a standard puck.

After the sample is mounted or the grease is applied for an addenda measurement, you may remove the assembly from the mounting station in the usual way and remove the calorimeter from the adapter.

8.2.5 Heat Capacity Cable and User Bridge

With a standard Heat Capacity system, the four-pin Lemo on the controller cable (part number 4085-100) is plugged into the system bridge board for reading the puck thermometer. With the Helium-3 system, the Helium-3 thermometer is read by using the standard user bridge board. Therefore, it is necessary to move the cable from the “P2” to the “P1” position on the back of the Model 6000 when going from standard Heat Capacity operations to Helium-3.

8.2.6 Hardware and Software Initialization

The following steps must be completed before performing a Heat Capacity measurement with the Helium-3 system:

1. Review the Helium-3 system operating procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to set up the Helium-3 hardware for a measurement.
2. Start up the Helium-3 software by selecting **Utilities >> Activate Option** in MultiVu and then select and activate **Helium3** in the **Available Options** panel.
3. Minimize the Helium-3 console, if you like. The Helium-3 software runs in the background, so the Helium-3 console does not have to be visible in the MultiVu interface while you work with the Helium-3 system.
4. Start up the Heat Capacity software by selecting **Utilities >> Activate Option** in MultiVu and then select and activate **Heat Capacity for He3**.

8.2.7 Performing Addenda and Sample Measurements

Once the software is running, there are very few procedural differences between running a measurement with a standard puck and a Helium-3 puck. All measurements begin by following the appropriate installation wizard from the Heat Capacity software. Please review the relevant sections of Chapters 6 and 7 for instructions on performing heat capacity measurements.

Other than the lower temperature range and the special handling of the Helium-3 insert—which is described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*—the measurement procedures are identical

8.2.8 Calibration Files

The calibrations for the Helium-3 calorimeter pucks reside in the `\TempCal\He3` directory. The Helium-3 calibrations are distinguished with names like `He3PuckXX.cal` rather than `PuckXX.cal`, where `XX` is the serial number of the puck. Also, the temperature range for the Helium-3 pucks covers the range from approximately 0.4 to 350 K, whereas the standard pucks cover the range from 1.9 to 400 K.

Section 8.5 discusses the calibration procedure in detail.

8.3 Measuring the Addenda

As with heat capacity measurements without the Helium-3 insert, you must first measure the heat capacity of the platform and grease (addenda) before measuring a sample. Please review the procedures in Chapter 6 before proceeding. Also, it is assumed that you are familiar with the Helium-3 probe-handling procedures described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*.

This section basically repeats the procedures in Chapter 6.

1. Verify that the Heat Capacity DSP cable is properly connected as described in Section 2.3.4.
2. Start the Helium-3 and Heat Capacity software as described in Section 8.2.6.
3. In the **Installation Wizards** tab in the Heat Capacity control center, select **Prepare Addenda Measurement**. Then follow the instructions to warm up and open the sample chamber.
4. Use the sample-mounting station as described in Section 8.2.4 to apply a small amount of grease to the Helium-3 calorimeter platform. As with the standard puck, Apiezon N Grease is preferred, but H Grease can be used if measurements are required above room temperature.
5. Place the protective cap back onto the puck, remove the puck from the mounting station and adapter, and install it onto the bottom end of the Helium-3 insert. Use the .050 hex Allen wrench to gently snug—but not tighten—the two hex Allen screws on the back of the sample-mounting plate to ensure good thermal contact to the mounting surface. Apply a small amount of Apiezon H Grease or Apiezon N Grease at the interface if necessary.
6. Locate and note the serial number on the Helium-3 calorimeter puck. The serial number is written on the top of the puck frame and is needed to start the measurement.
7. Follow the procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to insert the probe into the PPMS sample chamber.
8. Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck.
9. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start an addenda measurement by selecting **Create New Addenda Table** in the **Measurement** tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Refer to Section 6.3.6.

Once the addenda measurement is complete, you may measure a sample by following the procedures in the following section.

8.4 Measuring Sample Heat Capacity

Please review the procedures in Chapter 7 before proceeding. Also, it is assumed that you are familiar with the Helium-3 probe-handling procedures described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*.

This section basically repeats the procedures presented in Chapter 7.

1. Verify that the Heat Capacity DSP cable is properly connected as described in Section 2.3.4.
2. Start the Helium-3 and Heat Capacity software as described in Section 8.2.6.
3. In the **Installation Wizards** tab in the Heat Capacity control center, select **Prepare Sample Measurement**. Then follow the instructions to warm up and open the sample chamber.
4. Use the sample-mounting station as described in Section 8.2.4 to mount a sample onto the grease on the Helium-3 calorimeter platform. The grease should have been applied prior to the addenda measurement.
5. Place the protective cap back onto the puck, remove the puck from the mounting station and adapter, and install it onto the bottom end of the Helium-3 insert. Use the .050 hex Allen wrench to gently snug—but not tighten—the two hex Allen screws on the back of the sample-mounting plate to ensure good thermal contact to the mounting surface. Apply a small amount of Apiezon H Grease or Apiezon N Grease at the interface if necessary.
6. Locate and note the serial number on the Helium-3 calorimeter puck. The serial number is written on the top of the puck frame and is needed to start the measurement.
7. Follow the procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to insert the probe into the PPMS sample chamber. Be very careful not to bump the end of the probe. The extra weight of the sample can easily break wires on the puck if bumped.
8. Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck.
9. Verify that the correct addenda table is selected.
10. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start a sample measurement by selecting **Measure Sample Heat Capacity vs Temperature** in the **Measurement** tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Refer to Section 7.3.9.

8.5 Calibrating the Helium-3 Calorimeter Puck

The calibration of the Helium-3 calorimeter puck, like the calibration of the standard calorimeter puck, consists of two separate parts, or passes. Without the Helium-3 insert, you must first remove the charcoal holder for the first pass of the calibration, and then reinstall it for the second pass. With the Helium-3 insert, you install a calibration fixture (part number 4092-624) for the first pass, and remove it for the second pass. Please review the procedures in Chapter 5 before proceeding.

If you will need to calibrate the thermometers for a specific puck in a magnetic field, refer to Chapter 5 for detailed instructions.

Also, it is assumed that you are familiar with the Helium-3 probe-handling procedures described in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual*.

8.5.1 Install the Calibration Fixture

To ensure proper thermal equilibrium, you must install the calibration fixture on the calorimeter puck before proceeding.

1. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 8.2.4.
2. Keep the puck on the sample-mounting station and keep the lever closed. Then carefully remove the protective cap from the puck.
3. Apply a small amount of Apiezon N Grease to the tip of the calibration fixture as shown in Figure 8-3. This grease is used to make thermal contact with the sample platform on the puck.

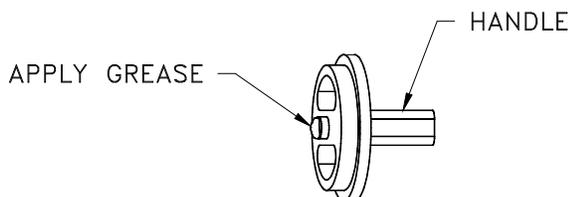


Figure 8-3. Applying Grease to Calibration Fixture

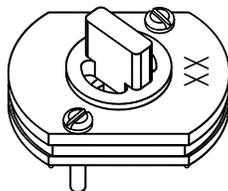


Figure 8-4. Calibration Fixture on Calorimeter Puck

4. Carefully insert the calibration fixture into the window on the calorimeter puck as shown in Figure 8-4. Note that the handle must be oriented diagonally to fit into the window. The tip with the grease should touch the sample platform. Twist the calibration fixture firmly to lock it into place.
5. Remove the puck and adapter from the sample-mounting station.

6. Remove the puck from the black plastic adapter, and then install the puck onto the bottom end of the Helium-3 insert. Do *not* use the handle on the calibration fixture to hold the puck; the fixture can slip out and detach the calorimeter platform.
7. Use the .050 hex Allen wrench to gently snug—but not tighten—the two hex Allen screws on the back of the sample-mounting plate to ensure good thermal contact to the mounting surface. Apply a small amount of Apiezon H Grease or N Grease at the interface if necessary.

8.5.2 Run the First Pass of the Puck Calibration

As with the calibration process for the standard puck, this pass is used to calibrate the thermometer on the Heat Capacity platform.

1. Verify that the Heat Capacity DSP cable is properly connected as described in Section 2.3.4.
2. Start the Helium-3 and Heat Capacity software as described in Section 8.2.6.
3. In the **Installation Wizards** tab in the Heat Capacity control center, select **Prepare New Puck Calibration**. Then follow the instructions to warm up and open the sample chamber.
4. Locate and note the serial number on the Helium-3 calorimeter puck. The serial number is written on the top surface of the puck and is needed to start the calibration.
5. Follow the procedures in the *Physical Property Measurement System: Helium-3 Refrigerator System User's Manual* to insert the probe into the PPMS sample chamber.
6. Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck. Select **He3Puck** as the type of puck you are calibrating.
7. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**. The **Puck Calibration (Pass 1)** dialog box opens.

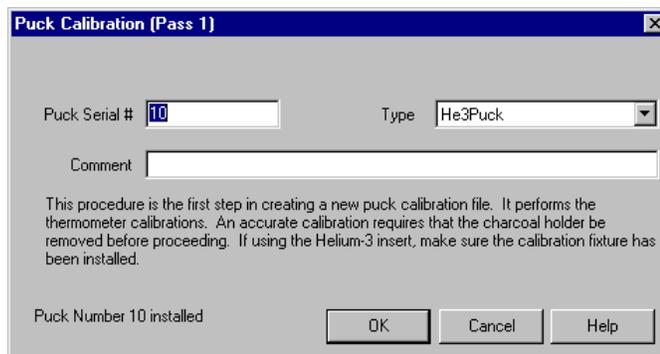


Figure 8-5. Puck Calibration (Pass 1) Dialog Box

8. Enter a comment, if desired, then select **OK**. The calibration begins, and the Measurement Status Viewer opens. The length of time the calibration runs varies from system to system. The first pass of the calibration will probably run for approximately 20 hours. The system will then warm up and lead you through the second pass of the calibration.

8.5.3 Remove the Calibration Fixture

Once the first pass is complete, the system warms up and prompts you to remove the calibration fixture. Proceed as follows:

1. Wait for the “Chamber is now flooding” message to appear in the **Setup for Pass 2 of Puck Calibration** dialog box. The message indicates that system conditions are correctly set for removing the Helium-3 insert from the sample chamber. If the system has been idle, you may need to press the **open chamber** button for this.
2. Stand on the step stool included with the Helium-3 option, and then remove the Helium-3 insert from the PPMS sample chamber and place it on the Helium-3 cart. Refer to the *Physical Property Measurement System: Helium-3 Refrigerator System User’s Manual* for detailed instructions.
3. Remove the calorimeter puck from the Helium-3 probe and mount it onto the sample-mounting station as described in Section 8.2.4.
4. Keep the puck on the sample-mounting station and keep the lever closed. Then remove the calibration fixture from the window on the calorimeter puck by twisting to loosen it and *very slowly* raising it out of the window. The chip must be securely held down by the vacuum suction of the mounting station.

CAUTION!

If the chip is not securely held down or you try to remove the calibration fixture too quickly, the chip will be pulled off with the fixture and destroyed.

5. Once the fixture is removed, place the protective cap back on the puck and remove the puck from the mounting station and adapter.
6. Install the calorimeter puck onto the bottom end of the Helium-3 insert as before.
7. Install the platform stabilizer plug by following the steps in Section 8.2.3.

8.5.4 Run the Second Pass of the Puck Calibration

1. Follow the procedure in the *Physical Property Measurement System: Helium-3 Refrigerator System User’s Manual* to reinsert the Helium-3 insert back into the sample chamber. If you notice condensation on the probe shaft, you may want to allow the shaft to become warm and dry before continuing. This will speed up the pump-out process.
2. Continue with the instructions in the installation wizard by purging the chamber, performing the puck test, and selecting a new data file, if desired.
3. Select **Finish**. The **Puck Calibration (Pass 2)** dialog box opens.

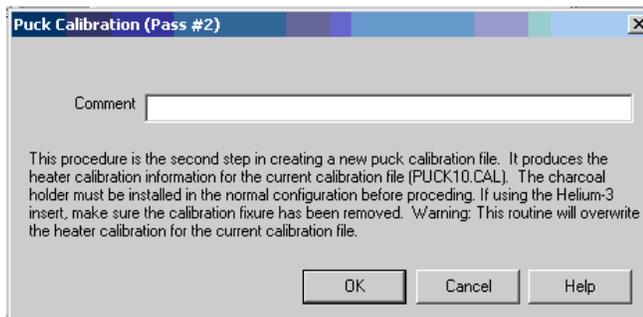


Figure 8-6. Puck Calibration (Pass 2) Dialog Box

4. Enter a comment, if desired, then select **OK**. The calibration begins, and the **Measurement Status Viewer** opens. The second pass of the calibration may take more time than the first pass, because when the High-Vacuum system is active, the temperature control is slower. The actual length of time the calibration runs varies from system to system.

When the second pass of the calibration is complete, the system warms up the sample chamber and prompts you to remove the puck. The `He3PuckXX.cal` file is then complete and ready for measurements.

Operation with the Dilution Refrigerator (DR) System

9.1 Introduction

This chapter contains the following information:

- Section 9.2 describes the special considerations and hardware required for measuring heat capacity with the PPMS DR option.
- Section 9.3 explains how to calibrate the DR calorimeter puck.
- Section 9.4 explains how to measure the addenda with the DR option.
- Section 9.5 explains how to measure the sample heat capacity with the DR option.

9.2 Special Considerations for Performing Heat Capacity Measurements with the Dilution Refrigerator Insert

The PPMS Dilution Refrigerator System (DR) option (Model P850) and the Heat Capacity option were designed to function together. Performing heat capacity measurements with the DR insert is similar to measuring without an insert and is almost identical to measuring with the Helium-3 insert (see Chapter 8). With the DR, heat capacity measurements can be performed over a temperature range from 0.05 K to 4 K, and at the highest fields available with a PPMS.

You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before you proceed with a corresponding measurement using the DR insert. As with standard pucks, before you measure the heat capacity of a sample, you must first measure the addenda to create a table. Then you can mount the sample on the puck and perform the measurement.

This section describes some of the key differences between heat capacity measurements that are performed with and without the DR insert.

9.2.1 Physics of Measuring Heat Capacity at Millikelvin Temperatures

The physics of measuring heat capacity at millikelvin temperatures is the same as that described in Chapter 1 of this manual. However, since the thermal conductivities and heat capacities of matter can be vastly different at 0.05 K than at 1 K, there are significant differences in construction details of a DR calorimeter as compared to the Helium-3 and Standard calorimeters.

Two physical effects are largely responsible for these differences. First, at millikelvin temperatures, there is the so-called Kapitza boundary resistance, where the thermal conductance at the interface between different materials is proportional to the temperature cubed. For example, for a metallic sample greased to a sapphire platform using N-grease, the thermal conductance between sample and the sapphire is approximately 20 microwatts per kelvin at 1 K, yet is only about 10 nanowatts per kelvin at 0.05 K. Because a successful heat capacity measurement requires a certain amount of thermal equilibrium between a sample and the thermometer and heater, this places constraints on the thermal conductance of the thermal link of the calorimeter.

The other important consideration is related to the large disparity between the heat capacities of different materials near 0.05 K and also the effect of magnetic field on heat capacity. Table 9-1 illustrates the enormous variety of heat capacities that different materials can exhibit at, for example, 0.1 K. Note that for a 10 mg sample, the heat capacity of different materials can vary by more than seven orders of magnitude. This representative list puts limits on the types and sizes of samples that can be measured and determines the permissible addenda heat capacity that is acceptable for a useful calorimeter.

Table 9-1 Examples of different types of specific heat in 10 mg samples at 0.1 K.

Physical Source of Heat Capacity	Functional Form	Example Heat Capacity at 0.1 K
Phonons	$C(T) \sim \beta T^3$	10 mg Sapphire: 0.001 nJ/K (extrap)
Electrons	$C(T) \sim \gamma T$	10 mg Gold: 3.5 nJ/K 10 mg Palladium: 88 nJ/K 10 mg Aluminum (normal, ~200 Oe): 50 nJ/K
Heavy Fermions	$C(T) \sim \gamma T$	10 mg PrInAg ₂ : ~15000 nJ/K
Structural tunneling transitions in disordered materials	$C(T) \sim aT^n$ ($1 < n < 2$)	10 mg Apiezon N grease: 1.6 nJ/K 10 mg Stycast 1266 epoxy: 3.1 nJ/K
Magnetic specific heat (Schottky Effect)	$C(T) = C_{\text{schottky}}(T, B)$	10 mg Manganin (Cu-13%Mn): ~10000 nJ/K

Variation of heat capacity with magnetic field can result from both nuclear and electronic energy level splitting in a magnetic field. The resulting Schottky heat capacity anomaly can cause the heat capacity of many materials to vary over several orders of magnitude as the field is changed. If the calorimeter frame is made of materials exhibiting a Schottky effect, the temperature control of the DR sample stage may be adversely affected in a magnetic field. Also, to avoid large variations in the heat capacity

of the platform, all the construction materials of the platform and wires must be chosen carefully to avoid excess Schottky heat capacity.

9.2.2 DR Calorimeter Puck

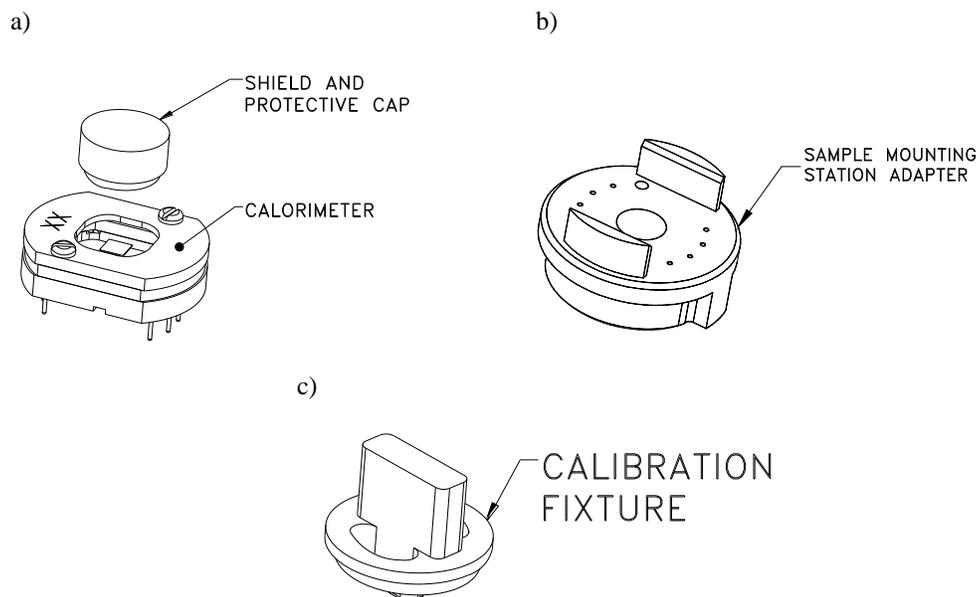


Figure 9-1. (a) Dilution Refrigerator Calorimeter puck (part number 4085-264), (b) Mounting Adapter (4092-625, and (c) Calibration Fixture (4091-624).

The calorimeter puck used on the DR insert is shown in Figure 9-1(a). It is similar in appearance to the frame assembly used with the standard Heat Capacity puck and the Helium-3 puck. The electrical connections and pin layout are identical and there is a locking cap like the Helium-3 puck. Also, the DR calorimeter adapts to the standard sample mounting station using the same adapter used by the Helium-3 calorimeter. However, because of the physics of thermal measurements at 0.05 K, there are also several significant differences between the DR calorimeter and the standard and Helium-3 designs.

9.2.2.1 USING SILVER INSTEAD OF COPPER

The metal frame of the DR calorimeters is made of silver rather than copper. This allows consistent temperature control at high magnetic fields because the heat capacity of silver does not depend on magnetic field down to 0.05 K. The heat capacity of copper at these temperatures, like many materials, has a strong field dependence from the Schottky effect.

Note: Both the DR calorimeter puck and the calibration fixture [Figure 9-1(c)] are made of silver and are then gold plated. With the gold plating, the parts can be easily confused with the corresponding gold-plated copper parts used with the Helium-3 insert (see Figure 8-1). Take precautions to avoid confusion if you also have a Helium-3 insert.

9.2.2.2 THERMAL LINK AND SUSPENSION

The thermal link between the sample platform and the calorimeter frame must be weak enough allow adequate thermal equilibrium to be achieved between the sample and platform during a relaxation measurement. At 0.05 K, the Kapitza boundary resistance limits thermal equilibration between different objects. To achieve reasonable thermal equilibrium between the sample and the platform, the thermal link to the platform should be about a factor of 10 smaller than the sample coupling to the platform. Hence, the thermal link to the bath was designed to be approximately 1 nanowatt per kelvin at 0.05 K. The fine wires needed to achieve this thermal stand-off are inadequate for mechanical support, so polymer supports are used for suspension.

9.2.2.3 THERMAL RADIATION

The frame and cap were also designed with special attention given to screening thermal radiation from outside the puck frame and cap. Because of the low thermal conductance of the thermal link, thermal radiation from the 2 K surroundings can warm the sample platform by several millikelvin. The puck and cap are design to greatly reduce radiative heating by preventing line-of-sight access to the sample platform during normal measurements.

9.2.2.4 THERMOMETER AND HEATER CONSTRUCTION

The resistance thermometer and heater are patterned directly on the underside of the sapphire sample platform. The thick-film materials used for the construction were selected for low Schottky heat capacity. The sensitivity of the thermometer has been tuned for maximum response in the 0.05 K to 4 K range. The calorimeter thermometer is useable up to above 20 K. This is considerably above the 4 K upper limit of the DR insert and can be reached if run on a Helium-3 insert or mounted on a standard puck chuck (see Figure 3-1).

9.2.3 Measurements in Magnetic Fields

As described in Chapters 5, 6, and 7, the thermometer used on the platform produces temperature errors of up to 20% in magnetic fields of several tesla when used at the lowest temperatures. This is also true for the DR platform thermometer. This magneto-resistance effect requires special handling to perform successful measurements in fields. The DR system thermometer has a correction built in for correcting temperature errors in the DR temperature control due to magnetic field. To obtain this correction for heat capacity measurements it is necessary to add magnetic field calibration data to the `.cal` file as described in Section 5.4.

Also, as described in Chapter 6, the heat capacity of the addenda changes with applied magnetic fields. However, depending on how large the heat capacity of your sample is compared to the addenda, it may or may not be necessary to account for this field dependence. In the case where the best possible accuracy is needed, you will need to measure the addenda for each of the corresponding fields of interest.

9.2.4 Vibration of the Sample Platform

Because of the extremely weak thermal link between the platform and calorimeter frame, heat sources as small as 1 pW can create unacceptable levels of heating of the platform. When this heating is not constant in time, the result will be large scatter in the resulting heat capacity measurements. Below about 0.5 K, vibrations of the DR and cryostat can produce substantial vibrational warming of the sample platform. The calorimeter puck has been designed for good rigidity to help address this. However, external vibrations can still produce unacceptable levels of heating on the platform. This may require that the PPMS cryostat be vibrationally isolated from the floor. Please contact Quantum Design for further assistance if you suspect vibrational heating of the calorimeter platform.

9.2.5 Helium Contamination in the Sample Chamber and Thermal Shorting

At DR temperatures, small amounts of helium contamination in the sample chamber can have substantial effects on heat capacity measurements.

9.2.5.1 THE HIGH VACUUM SYSTEM: CRYOPUMPS AND CHARCOAL

The Heat Capacity option always performs measurements under vacuum conditions to minimize thermal conductance through the gas. At DR temperature, there are other effects beside gas conductance that become important, including the heat capacity and thermal conductance of adsorbed helium films. To minimize helium gas and adsorbed superfluid helium films on surfaces in and around the calorimeter puck, the DR software is designed to isolate the sample chamber space from the main chamber cryopump once high vacuum has been achieved. This is normally done at about 20 K during the cooldown of the DR insert. Sealing the chamber prevents back-streaming of helium from the sorption pumps in the cryopump. To remove additional helium from the sample chamber space once it is sealed, there are two additional charcoal sorption pumps located within the sample chamber. One is composed of charcoal cloth and it is mounted on the underside of the screw-on end-cap on the DR insert. This charcoal is maintained at about 1.8 K during normal DR operation. The other charcoal sorption pump is also composed of charcoal cloth and it is located in the underside of the protective cap on the calorimeter puck (see Fig 9-1).

9.2.5.2 SYMPTOMS OF HELIUM CONTAMINATION: ENHANCED HEAT CAPACITY AND THERMAL SHORTING

Samples with a lot of surface area (on a microscopic level) can have a high affinity for helium. In some cases, the result is a slightly larger measured heat capacity. Also, you may find that the measured heat capacity of your sample gets larger over time as more helium adsorbs on it. Another possibility is that the relaxation curves appear to be poorly fit by the thermal model at the lowest temperature.

Another symptom of helium contamination is a Pass 2 calorimeter calibration that fails at or very close to 1.0 K. This failure is caused by an interaction between the polymer supports (small polyimide tubes) described in Section 9.2.2.2 and superfluid helium films. Excess helium in the chamber can create an almost perfect thermal short between the platform and the calorimeter frame. This thermal short appears suddenly below 1 K and gradually goes away again below 0.7 K. So there is no effect above 1.1 K and no effect below 0.5 K. The effect is so dramatic, that it can easily be misdiagnosed as an electronics failure or software bug.

9.2.5.3 CORRECTING HELIUM CONTAMINATION: REGENERATE ALL CHARCOAL

If you suspect helium contamination, you should do the following:

1. Regenerate the sample chamber cryopump. Follow the procedures for your particular system (PPMS or Dynacool).
2. Make sure the charcoal on both the calorimeter cap and the end of the DR insert is fully regenerated. To do this, you will need to remove these two caps and heat them to 150° to 200°C for 1 to 2 hours. This can be done with any oven or hot plate. Before baking, wipe off any excess oils or grease from the metal parts to prevent the contamination from running into the charcoal and clogging the pores. If you suspect that the charcoal in either the calorimeter cap or the DR end-cap has become contaminated with oil, melted grease, or solvents, then the charcoal will need to be replaced. Never bake the entire calorimeter puck.

If you suspect helium contamination from a leak, either in your sample chamber or in the DR insert, please contact Quantum Design for assistance.

9.2.6 Using the Sample-Mounting Station

When setting up a puck calibration, addenda measurement, or sample measurement, it is necessary to use the sample-mounting station to immobilize the platform.

A special adapter [Figure 1(b)] is provided for the purpose of allowing you to use the standard sample-mounting station with the DR calorimeter pucks. The following steps describe how you use the sample-mounting station.

1. Remove the calorimeter puck from the DR insert if the puck is still on the insert.
2. Attach the calorimeter puck to the sample-mounting station adapter. It is a good idea to keep the protective cap on during this step. The eight pins should slip easily into the eight holes and the puck should fit nicely between the two raised sections of the adapter.
3. Optionally, you can use the two long screws through the back side of the adapter to snugly hold the calorimeter onto the adapter. For convenience, coat the screws with H grease before you insert them into the holes to keep the screws from falling out.
4. Insert the assembly onto the mounting station and completely close the lever arm as you would with a standard puck.
5. After the sample is mounted or the grease is applied for an addenda measurement, you may remove the assembly from the mounting station in the usual way and remove the calorimeter from the adapter.

9.2.7 DR Heat Capacity Cable/Filter Box

When the DR is inserted into the PPMS sample chamber for a measurement, the insert is connected to the CM-E Heat Capacity Module using the DR Heat Capacity Cable/RF Filter Box (part number 3091-620), shown in Figure 9-2. This cable assembly has an RF filter box integrated into the end that plugs into the DR insert. The filters eliminate stray RF currents that would otherwise be conducted into the insert from the outside and cause self-heating of the thermometers and anomalous temperature readings. Connect the 15-pin D-shell connector to the CM-E module and connect the filter box to the DR insert at the 10-pin Fischer connector labeled “SAMPLE” (lower connector on the right side of the insert).

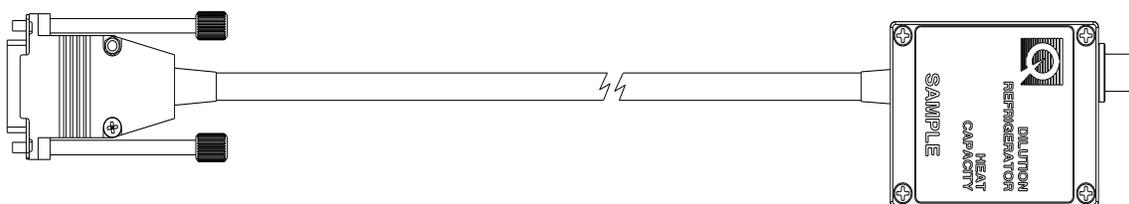


Figure 9-2. DR Heat Capacity Cable/RF Filter Box

9.2.8 Software Initialization

The following steps must be completed before performing a Heat Capacity measurement with the DR system:

1. Review the DR system operating procedures in the PPMS DR User's Manual to set up the DR hardware and make sure the DR gas handling system is connected.
2. Start the MultiVu software if it is not already running and wait for MultiVu to recognize the DR system. This will be indicated by the presence of the DR Option Control Console dialog that pops up shortly after starting MultiVu. Do not connect the TEMPERATURE cable or the SAMPLE cable to the DR insert yet. It is easier to safely handle the DR insert if these connections are made after inserting it into the PPMS sample chamber.
3. Start up the Heat Capacity software by selecting **Utilities >> Activate Option** in PPMS MultiVu and then select and activate **Heat Capacity for DR**. (If the **Heat Capacity for DR** option is not available in the list, you will need to start over and reinstall the Heat Capacity option software as described in Section 2.3.1.)

9.3 Calibrating the DR Calorimeter Puck

The calibration of the DR calorimeter puck, like the calibration of the standard calorimeter puck, consists of two separate parts, or passes. With the standard HC system, you must first remove the charcoal holder for the first pass of the calibration, and then reinstall it for the second pass. With the DR insert, you install the calibration fixture [Figure 9-1(c)] for the first pass, and remove it for the second pass. Please review the procedures in Chapter 5 before proceeding.

To calibrate the puck for measuring in magnetic fields, see the Helium-3 example described in Section 5.4.1.

You only need to follow these calibration procedures once for a given puck. If your DR puck has already been calibrated you may skip this section.

It is assumed that you are familiar with the DR probe-handling procedures described in the PPMS DR System User's Manual.

9.3.1 Install the Calibration Fixture

To ensure proper thermal equilibrium, you must install the calibration fixture [Figure 9-1(c)] on the calorimeter puck before proceeding.

Note: If you also have a Helium-3 inert, do not confuse the calibration fixture described in this section with the one used for Helium-3 calibrations. The one used here is made of silver (see Section 9.2.2.1 above) and is a slightly different shape.

1. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 9.2.5.
2. Carefully remove the radiation cap from the puck and apply a small amount of Apiezon N Grease to the tip of the calibration fixture as shown in Figure 9-3. This grease is used to achieve thermal equilibrium between the fixture and the calorimeter platform.

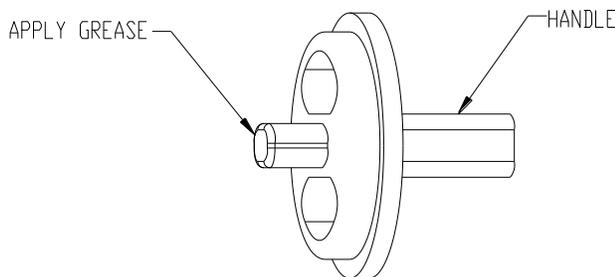


Figure 9-3. Applying Grease to Calibration Fixture

- Carefully insert the calibration fixture into the window on the calorimeter puck as shown in Figure 9-4. Note that the handle must be oriented diagonally to fit into the window. The tip with the grease should touch the sample platform. Twist the calibration fixture firmly enough to lock it into place.

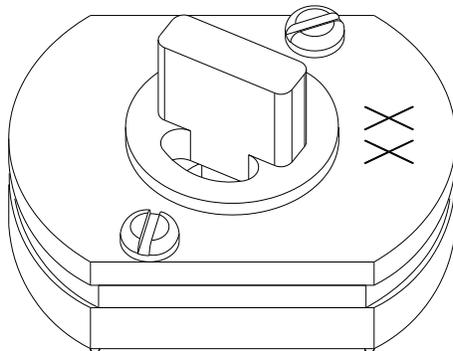


Figure 9-4. Calibration Fixture on Calorimeter Puck

- Remove the puck and adapter from the sample-mounting station.
- Remove the puck from the sample-mounting adapter, and follow the instructions in Section 4.4.1 “Mounting and Un-Mounting Samples” in the PPMS DR User’s Manual to attach the calorimeter to the DR insert. Do not use the handle in the calibration fixture to hold the puck; the fixture can slip out and detach the calorimeter platform.
- Be sure to note the serial number on the calorimeter puck before installing the radiation shield on the end of the DR insert. The serial number is written on the top surface of the puck and is needed to start the calibration.

9.3.2 Run the First Pass of the Puck Calibration

As with the calibration process for the standard puck, this pass is used to calibrate the thermometer on the calorimeter platform with respect to the DR system thermometer.

- Verify that the software is activated as described above in Sections 9.2.6 and 9.2.7.
- In the **Installation Wizards** tab in the Heat Capacity control center in MultiVu, select **Prepare New Puck Calibration**. Then follow the instructions to warm and open the sample chamber.
- Follow the procedures in Section 4.4.2 “Inserting the DR Probe” in the PPMS DR User’s Manual to insert the DR into the PPMS sample chamber. Verify that the Heat Capacity Cable (Figure 9-2) is properly connected to the DR.
- Continue with the instructions in the installation wizard by purging the chamber and entering the serial number of the puck. Select **DRPuck** as the type of puck you are calibrating.
- Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**. The **Puck Calibration (Pass 1)** dialog box opens.

Chapter 9

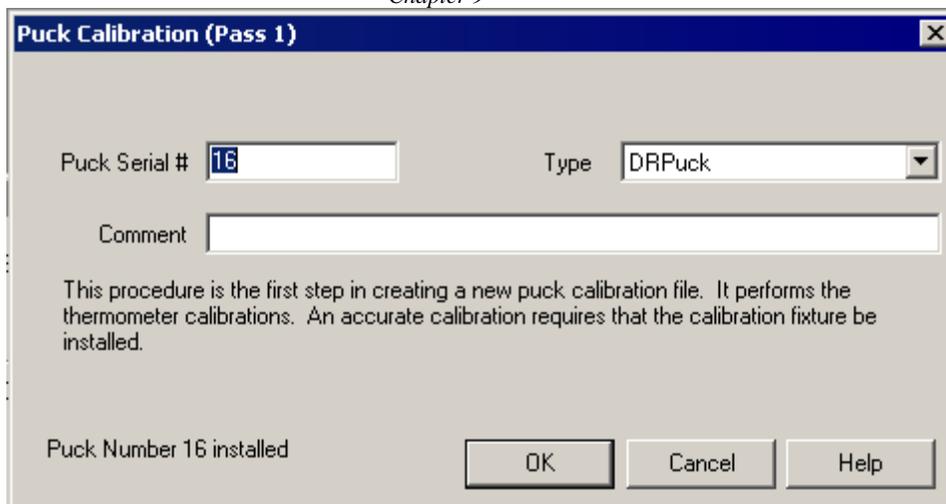


Figure 9-5. **Puck Calibration (Pass 1)** Dialog Box

6. Enter a comment, if desire, then select **OK**. The calibration begins, and the measurement Status View opens. The length of the calibration run varies from system to system. The first pass of the calibration will probably run for approximately 12 hours. The system will then automatically warm up and guide you through the second part of the calibration.

9.3.3 Remove the Calibration Fixture

Once the first pass is complete, the system warms up and prompts you to remove the calibration fixture. Proceed as follows:

1. Wait for the “Chamber is now flooding” message to appear in the **Setup for Pass 2 of Puck Calibration** wizard page. This message indicates that the system is ready for removing the DR insert from the sample chamber.
2. Follow the procedures in Section 4.4.4 “Removing the Dilution Refrigerator Probe” and Section 4.4.5 “Remove the Sample Mount” in the PPMS DR User’s Manual to remove the calorimeter puck from the DR insert.
3. Mount the calorimeter onto the sample mounting station as described in Section 9.2.5. It is critical that the vacuum be applied to hold the platform down during the next step.
4. Keep the puck on the sample-mounting station and keep the lever closed. Then remove the calibration fixture from the window on the calorimeter puck by twisting to loosen it and *very slowly* raising it out of the window. The chip must be securely held down by the vacuum suction of the mounting station.

CAUTION!

If the chip is not securely held down or you try to remove the calibration fixture too quickly, the chip will be pulled off with the fixture and destroyed.

5. Once the fixture is removed, place the calorimeter radiation cap back on the puck and remove the puck from the mounting station and adapter
6. Again, follow the instructions in Section 4.4.1 “Mounting and Un-Mounting Samples” in the PPMS DR User’s Manual to attach the calorimeter to the DR insert.
7. Again, follow the procedures in Section 4.4.2 “Inserting the DR Probe” in the PPMS DR User’s Manual to insert the DR into the PPMS sample chamber and connect the Heat Capacity Cable.

9.3.4 Run the Second Pass of the Puck Calibration

1. Follow the procedures in Section 4.4.2 “Inserting the DR Probe” in the PPMS DR User’s Manual to insert the DR into the PPMS sample chamber. If you notice condensation on the probe shaft, you may want to allow the shaft to become warm and dry before continuing.
2. Continue with the instructions in the installation wizard by purging the chamber, performing the puck test, and verifying the data file.
3. Select Finish. The Puck Calibration (Pass 2) dialog opens.

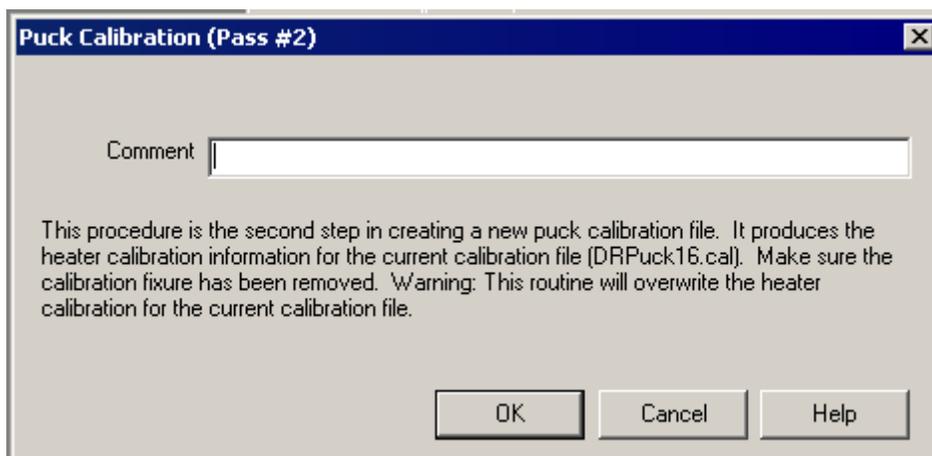


Figure 9-6. **Puck Calibration (Pass 2)** Dialog Box

4. Select **OK**. The calibration begins, and the **Measurement Status Viewer** opens. The second pass of the calibration will take about the same time as the first pass (about 12 hours). The actual length of the calibration will vary from system to system.
5. When the second pass of the calibration is complete, the system automatically warms up the sample chamber and prompts you to remove the puck. The .cal file is selected and ready for measurements.

In addition to calibration data, the pass 2 procedure also creates an addenda table. This addenda table can be used for performing a sample measurement.

Chapter 9

9.4 Measuring the Addenda

As with heat capacity measurements without the DR insert, you must first measure the heat capacity of the platform and grease (addenda) before measuring a sample. This procedure is only necessary if there are no previously acquired addenda tables, including the one created during the calibration, that are suitable for your sample. Please review the procedures in Chapter 6 before proceeding. It is assumed that you are familiar with the DR probe-handling procedures described in the PPMS DR System User's Manual.

This section basically repeats the procedures in Chapter 6, modified for the DR insert.

1. Verify that the software is activated as described above in Sections 9.2.6 and 9.2.7.
2. In the **Installation Wizards** tab in the Heat Capacity control center in MultiVu, select **Prepare Addenda Measurement**. Then follow the instructions to warm and open the sample chamber.
3. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 9.2.5.
4. Carefully remove the radiation cap from the puck and apply a small amount of Apiezon N Grease to the DR calorimeter platform.
5. Place the radiation cap back onto the puck, remove the puck and adapter from the sample-mounting station.
6. Remove the puck from the sample-mounting adapter, and follow the instructions in Section 4.4.1 "Mounting and Un-Mounting Samples" in the PPMS DR User's Manual to attach the calorimeter to the DR insert.
7. Be sure to note the serial number on the calorimeter puck before installing the radiation shield on the end of the DR insert.
8. Follow the procedures in Section 4.4.2 "Inserting the DR Probe" in the PPMS DR User's Manual to insert the DR insert into the PPMS sample chamber. Verify that the Heat Capacity Cable (Figure 9-2) is properly connected to the DR.
9. Continue with the instructions in the installation wizard by purging the chamber and verifying the serial number of the puck.
10. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start an addenda measurement by selecting Create New Addenda Table in the Measurement tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Again, refer to Chapter 6.

Once the addenda measurement is complete, you may measure a sample by follow the procedures in the following section.

9.5 Measuring Sample Heat Capacity

Please review the procedures in Chapter 7 before proceeding. Also, it is assumed that you are familiar with the DR probe-handling procedures described in the PPMS DR System User's Manual.

This section basically repeats the procedures in Chapter 7, modified for the DR insert.

1. Verify that the software is activated as described above in Sections 9.2.6 and 9.2.7.
2. In the **Installation Wizards** tab in the Heat Capacity control center in MultiVu, select **Prepare Sample Measurement**. Then follow the instructions to warm and open the sample chamber.
3. Mount the calorimeter puck you want to calibrate onto the sample-mounting station as described in Section 9.2.5.
4. Carefully remove the radiation cap from the puck and attach the sample to the grease on the calorimeter platform.
5. Place the radiation cap back onto the puck and remove the puck and adapter from the sample-mounting station.
6. Remove the puck from the sample-mounting adapter, and follow the instructions in Section 4.4.1 "Mounting and Un-Mounting Samples" in the PPMS DR User's Manual to attach the calorimeter to the DR insert.
7. Be sure to note the serial number on the calorimeter puck before installing the radiation shield on the end of the DR insert.
8. Follow the procedures in Section 4.4.2 "Inserting the DR Probe" in the PPMS DR User's Manual to insert the DR insert into the PPMS sample chamber. Verify that the Heat Capacity Cable (Figure 9-2) is properly connected to the DR.
9. Continue with the instructions in the installation wizard by purging the chamber and verifying the serial number of the puck, and selecting the correct addenda table. The correct addenda table may already be selected from a previous measurement.
10. Wait for the puck test to be successfully completed, and then select a data file, and select **Finish**.

You may now start a sample measurement by selecting **Measure Sample Heat Capacity vs Temperature** in the **Measurement** tab in the Heat Capacity control center or by writing a sequence with the equivalent sequence command. Again, refer to Chapter 7.

Operation with the VersaLab System

10.1 Introduction

This chapter contains the following information:

- Section 10.2 describes the procedures for installing the Heat Capacity Option for the first time.
- Section 10.3 discusses how using the Heat Capacity Option with VersaLab is different from using it on a standard PPMS system.

10.2 Installing the Hardware and Software for VersaLab

This section describes the first-time installation of a new Heat Capacity Option for VersaLab. For standard PPMS systems, follow Section 2.3 instead.

Detailed descriptions of the hardware are contained in Chapter 3. VersaLab-specific hardware is described in this chapter.

10.2.1 Install the Software

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

1. Install VersaLab MultiVu version 0.9.17 or higher by running setup.exe from the installation package.

Note: The VersaLab system uses different version numbers for the MultiVu software than for the Standard PPMS.

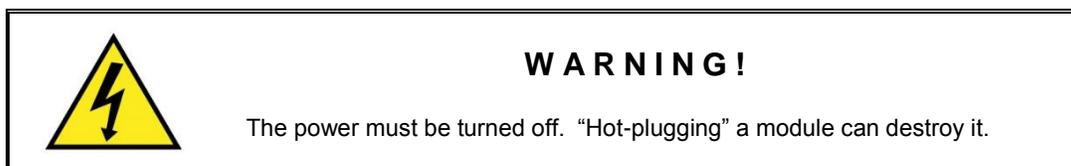
2. Install Heat Capacity version 3.6.0 or later.

10.2.2 Install the CM-E Heat Capacity Controller Module

The control electronics for the Heat Capacity Option are contained in the Model CM-E Heat Capacity Module. See Section 3.2.3 for a drawing and a detailed description of this module. This controller normally resides in one of the integrated module bays on VersaLab and may already have been installed.

If the Heat Capacity Module is not already installed, then do this now:

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



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

10.2.3 Connect the Controller Cable

A single controller cable (3085-110) connects the "Calorimeter JE-1" port on the CM-E Heat Capacity Module to the gray Lemo user port on the cryostat. Figure 10-1 shows this cable. The pin routing for the cable is identical to the standard controller cable and is given in Table 3-1.

Note: The "Puck Therm" (pins 11 thru 14) is not connected for VersaLab.

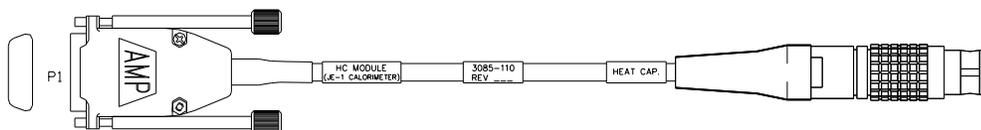


Figure 10-1. Controller Cable for VersaLab Heat Capacity.

10.2.4 Setup Sample Mounting Station

The sample mounting station is a tool to facilitate mounting samples onto the calorimeter puck without damaging the delicate wires suspending the sample platform. For a full description of the mounting station, see Section 3.2.2. To setup the mounting station, follow the instructions in Section 2.3.3.

10.2.5 Quick Start with VersaLab

Once the Heat Capacity Option is installed, follow the instruction in Section 2.4 for quick introduction to performing a first measurement (of addenda) on your VersaLab.

10.3 Special Considerations for Performing Heat Capacity Measurements using VersaLab

The Heat Capacity Option for VersaLab uses the same calorimeter pucks (Section 3.2.1), measurement software (Chapter 4), and measurement electronics (Section 3.2.3) as the standard PPMS Heat Capacity Option. Like the standard PPMS Heat Capacity measurement system, the VersaLab Heat Capacity Option is limited in temperature and field range by the capabilities of the host system. For VersaLab, heat capacity measurements can be performed over a temperature range from 50 to 400 K, and at magnetic fields up to 3 T.

You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before performing Heat Capacity measurements using VersaLab.

This section discusses how using the Heat Capacity Option with VersaLab is different from a using it on a standard PPMS system.

10.3.1 Calorimeter Puck

The VersaLab calorimeter puck is identical to the Standard PPMS Heat Capacity puck as described in Section 3.2.1. In fact, if the puck was calibrated using a model CM-E controller on a standard PPMS, both the puck and calibration file can be used on a VersaLab system.

Other than the limited temperature and field range of the VersaLab as compared to a PPMS, the only difference between the heat capacity options on these system is the use of the puck thermometer in the calorimeter. This auxiliary thermometer, which located inside the puck base, is normally used for faster temperature control. On the VersaLab, there is little or no benefit to using this thermometer, so it is not used. The puck thermometer is included only to maintain compatibility.

10.3.2 High Vacuum and Contact Baffle

With VersaLab, a thermally isolating vacuum is provided in the sample chamber using the built-in cryopump. The contact baffle assembly is included to provide shielding from thermal radiation. This reduces thermal offsets between the calorimeter puck and the chamber block when the chamber is at high vacuum and at either high or low temperatures. The contact baffle is shown in Figure 3-8. For all heat capacity measurements and calibrations, the contact baffle is screwed onto the VersaLab baffle assembly. This baffle assembly is the same as that shown in Figure 3-7, except that it is shorter than the standard PPMS model to match the length of the VersaLab sample chamber.

Since the VersaLab sample chamber does not go below about 50 K, it is not necessary to remove the charcoal assembly from the contact baffle during any part of the puck calibration procedure (see Chapter 5). In fact, the charcoal is not needed at all for high vacuum operations on VersaLab as long as helium exchange gas is used for chamber operations. As a result, unlike with a standard PPMS, both pass 1 and pass 2 of a puck calibration procedure can be done without opening the sample chamber.

10.3.3 Heat Capacity Software

The software used for the VersaLab Heat Capacity Option is the same as other Quantum Design Heat Capacity Options. The installation directory for the VersaLab software is `\QdVersaLab\`. VersaLab-specific installation subdirectories can be found in Table 4-1. For example, calibration files for the VersaLab pucks can be found in the directory `\QdVersaLab\HeatCapacity\TempCal\VersaLab\`. See Chapter 4 for a description of the Heat Capacity software features and options.

Operation with the DynaCool System

11.1 Introduction

This chapter contains the following information:

- Section 11.2 describes the procedures for installing the Heat Capacity Option for the first time.
- Section 11.3 discusses how using the Heat Capacity Option with DynaCool is different from using it on a standard PPMS system.

11.2 Installing the Hardware and Software for DynaCool

This section describes the first-time installation of a new Heat Capacity Option for DynaCool. For standard PPMS systems, follow Section 2.3 instead.

Detailed descriptions of the Heat Capacity hardware are contained in Chapter 3. DynaCool-specific hardware is described in this chapter.

11.2.1 Install the Software

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

1. Install the latest version of the DynaCool software package by running the installation. This will install the latest version of MultiVu.
2. Install the latest Heat Capacity option software from the Heat Capacity installer.

11.2.2 Install the CM-E Heat Capacity Controller Module

The control electronics for the Heat Capacity Option are contained in the Model CM-E Heat Capacity Module. See Section 3.2.3 for a drawing and a detailed description of this module. This controller normally resides in one of the module bays on the DynaCool CAN Module Bay on the side of the cryostat and may already have been installed.

If the Heat Capacity Module is not already installed, then do this now:

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



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

11.2.3 Connect the Controller Cable

A single controller cable (3085-110) connects the "Calorimeter JE-1" port on the CM-E Heat Capacity Module to the gray Lemo user port on the right side of the DynaCool cryostat. Figure 11-1 shows this cable. The pin routing for the cable is identical to the standard controller cable and is given in Table 3-1.

Note: The "Puck Therm" (pins 11 thru 14) is not connected for DynaCool.

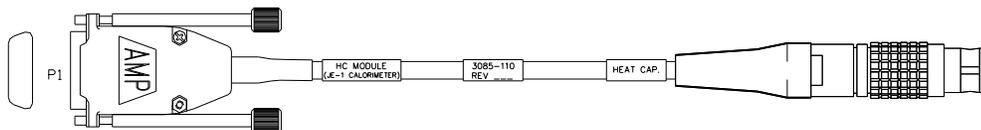


Figure 11-1. Controller Cable for DynaCool Heat Capacity.

11.2.4 Setup Sample Mounting Station

The sample mounting station is a tool to facilitate mounting samples onto the calorimeter puck without damaging the delicate wires suspending the sample platform. For a full description of the mounting station, see Section 3.2.2. To setup the mounting station, follow the instructions in Section 2.3.3.

11.2.5 Quick Start with DynaCool

Once the Heat Capacity Option is installed, follow the instruction in Section 2.4 for quick introduction to performing a first measurement (of addenda) on your DynaCool.

11.3 Special Considerations for Performing Heat Capacity Measurements using DynaCool

The Heat Capacity Option for DynaCool uses the same calorimeter pucks (Section 3.2.1), measurement software (Chapter 4), and measurement electronics (Section 3.2.3) as the standard PPMS Heat Capacity Option. Like the standard PPMS Heat Capacity measurement system, the DynaCool Heat Capacity Option is limited in temperature and field range by the capabilities of the host system. Without any optional refrigerator insert, DynaCool can perform heat capacity measurements over a temperature range from 1.8 to 400 K, and at magnetic fields limited only by your DynaCool magnet.

You should be completely familiar with the contents of Chapter 6, “Creating and Using Addenda Tables,” and Chapter 7, “Heat Capacity Measurement,” before performing Heat Capacity measurements using DynaCool.

This section discusses how using the Heat Capacity Option with DynaCool is different from a using it on a standard PPMS system.

11.3.1 Calorimeter Puck

The DynaCool calorimeter puck is identical to the Standard PPMS Heat Capacity puck as described in Section 3.2.1. In fact, if the puck was calibrated using a model CM-E controller on a standard PPMS, both the puck and calibration file can be used on a DynaCool system.

The only difference between the heat capacity options on these system is the use of the puck thermometer in the calorimeter. This auxiliary thermometer, which located inside the puck base, is normally used for faster temperature control on a standard PPMS. On the DynaCool, there is little or no benefit to using this thermometer, so it is not used. The puck thermometer is included only to maintain compatibility.

11.3.2 Heat Capacity Software

The software used for the DynaCool Heat Capacity Option is the same as other Quantum Design Heat Capacity Options. The installation directory for the DynaCool software is `\QdDynaCool\`. DynaCool -specific installation subdirectories can be found in Table 4-1. For example, calibration files for the DynaCool pucks can be found in the directory `\QdDynaCool\HeatCapacity\TempCal\Standard\`. See Chapter 4 for a description of the Heat Capacity software features and options.

Hardware Troubleshooting and Maintenance

12.1 Introduction

This chapter contains the following information:

- Section 12.2 explains how to use the puck adjustment tool.
- Section 12.3 explains how to clean pucks.
- Section 12.4 explains how to replace or repair a damaged puck frame.
- Section 12.5 explains how to test the calibration of the user and system thermometers.
- Section 12.6 explains what to do if the vacuum pump for the sample-mounting station produces excess noise.

12.2 Loose Puck: Using the Adjustment Tool

The puck adjustment tool (Figure 12-1) adjusts the tension in the chuck fingers so that the fingers maintain solid thermal contact with the heater block located at the bottom of the sample chamber. Solid thermal contact between the chuck fingers and the heater block is especially important for high-vacuum applications, such as heat capacity measurements.

The puck adjustment tool consists of two metal cylinders. In Figure 12-1, cylinder 1 is the finger spreader, and cylinder 2 is the finger contractor and the test cutout. The finger spreader and the finger contractor adjust the tension of the chuck fingers. The test cutout, which has the same dimensions as the cutout in the heater block, tests how well the chuck fingers will contact the heater block.

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

Another indicator of a loose puck is when it takes an unusually long time for the platform temperature to stabilize at a new temperature. For more information on the drift rate, see Section 7.6.

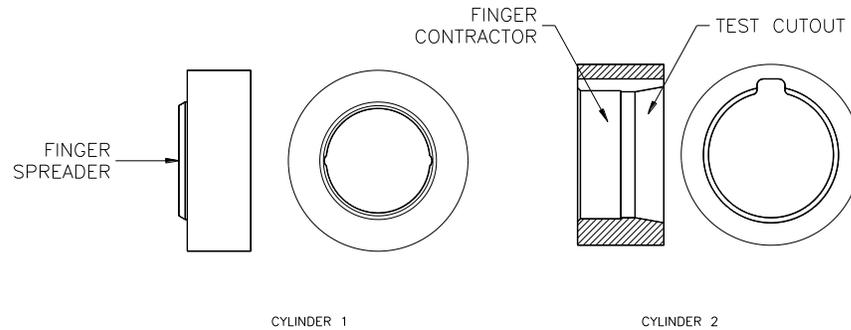


Figure 12-1. Puck Adjustment Tool

Complete the following steps to use the puck adjustment tool:

1. Slide the thermal radiation shield over the top of the puck.
2. Place the puck on the finger spreader. Refer to Figure 12-1. Turn the puck until the screw heads on the bottom of the puck line up with the grooves inside the finger spreader. Press the puck downward and continue pressing until all chuck fingers touch the base of the finger spreader. When all fingers touch the base of the spreader, the spreader evenly applies radial force to the fingers, pushing them outward and slightly beyond their optimal location.
3. Remove the puck from the finger spreader.
4. Place the puck inside the finger contractor. Refer to Figure 12-1. Press straight down on the puck and continue pressing until you press the puck completely into the finger contractor. When the entire chuck is in the contractor, the contractor evenly applies force to the outside of the fingers, pushing them inward. The contractor pushes the fingers—regardless of external wear or variations on the puck—so that the fingers obtain their optimal location.
5. Remove the puck from the finger contractor.
6. Place the puck inside the test cutout. Refer to Figure 12-1. Verify that the puck fits easily but snugly in the test cutout.

12.3 Cleaning Pucks

When you mount samples, you should work carefully to avoid getting grease on the sides or the underside of the sample holder platform. However, when cleaning is necessary, please use the following guidelines.

12.3.1 Degreasing Using a Cotton Swab

To avoid spreading grease on to the sides and underside of the sample platform, it is helpful to routinely remove the grease from the top of the platform after removing a sample and while the puck is still mounted on the sample-mounting station. To remove the grease, gently wipe the surface with a cotton swab moistened with a degreasing agent such as Toluene or 1,1,1-Trichloroethane (TCE). Take care not to touch the wires during this process, as they are very fragile. Also, do not use excessive

amounts of Toluene or TCE because the excess will be pulled into the small vacuum pump, possibly damaging the rubber diaphragms. Finally, avoid leaving behind cotton fibers on the wires or platform.

CAUTION!

Consult the manufacturer's health safety information accompanying both Toluene and TCE. Avoid skin contact and inhalation of vapors.

12.3.2 Degreasing the Entire Frame

If grease has spread to the sides and underside of the sample platform, it may be necessary to degrease the entire frame. Remove the frame from the puck by following step 1 in Section 12.4.1. Submerge the frame in a warm bath of soapy water for 15-30 minutes. Alternatively you can submerge the frame in a room-temperature bath of Toluene or TCE for approximately 5 minutes but no longer than 15 minutes. Rinse the frame in a bath of isopropyl alcohol and let it dry. Follow steps 2 and 3 in Section 12.4.1 to reattach the frame to the puck.

CAUTION!

The puck frame and sample platform are constructed using epoxy. Do not attempt to use any types of solvents not explicitly mentioned in this manual, because they may dissolve the epoxy and permanently damage the frame. Also, do not attempt to use an ultrasonic cleaner.

CAUTION!

This procedure should not be used with DR puck frames. DR calorimeters have internally greased joints that are required for operation. Degreasing agents could remove this grease.

12.4 Broken Puck Wire

A puck is unusable if any of the wires on it breaks. A broken wire is usually indicated by a failed puck test in an installation wizard in the Heat Capacity software.

12.4.1 Replacing the Puck Frame

The Heat Capacity puck is designed so that only part of it needs to be replaced in the event that the puck wires break or the sample platform is damaged. The standard Heat Capacity system is shipped with an additional puck frame for puck repairs. Broken frames can be reworked for a nominal charge, or you can purchase additional spare frames by contacting Quantum Design.

Complete the following steps to replace the puck frame:

1. Remove the two screws located on the bottom of the puck frame, and then remove the puck frame by carefully unplugging it from the eight sockets used for electrical contact. Save the screws.
2. Apply a small amount of Apiezon H Grease to the back of the new frame to ensure adequate thermal contact when the frame is attached to the chuck of the puck. Use just enough grease to make thermal contact, but not so much that an excess amount squeezes out when you assemble the parts.
3. Insert the eight pins of the new frame into the sockets through the top of the body of the puck. The guide pin ensures proper orientation of all parts. Locate the screws you removed in step 1. Use the screws to attach the frame snugly to the puck.
4. Calibrate the puck. Refer to Chapter 5.

12.4.2 Repairing a Damaged Puck Frame

Quantum Design does not recommend that you attempt to repair a broken puck frame. A spare frame is provided to minimize downtime while the broken frame is repaired by Quantum Design personnel. However, if you are compelled to attempt a repair yourself, this section provides some tips. Attempting to repair such a fragile device requires a steady hand and an expertise in soldering fine structures.

For the following procedures, you need tweezers and a soldering iron with a fine tip. You may also need to use a stereo microscope or a lighted magnifying glass.

If a single wire breaks loose from an eight-wire puck frame, you may try a couple different repairs. The simplest thing to do is to short the loose end of the wire to the remaining good wire at the pin rather than at the sample platform. This means that only a three-wire contact is made to the thermometer or heater. A more daring technique is to attempt to solder the wire back into place. Both techniques require that the puck be calibrated again. For these procedures, you need tweezers and a soldering iron with a fine tip.

Complete the following steps to bypass the loose end of the frame to the neighboring pin:

1. Remove the two screws that attach the wire guard to the puck frame, and then remove the wire guard. Save the screws.
2. Place the puck in the sample-mounting station. Turn on the small vacuum pump that is attached to the mounting station and then close the puck interlock arm. Verify that the vacuum holds the sample platform snugly on the platform holder.
3. If your soldering iron has a temperature adjustment, choose the lowest setting that will still melt the solder.

4. Use tweezers to locate the loose end of the broken wire. Solder the loose end to the neighboring pin as shown in Figure 12-2. Care must be taken to ensure that the wire already soldered to that pin remains intact.
5. Place the wire guard on top of the puck frame. Locate the two screws you removed in step 1. Use the screws to attach the wire guard to the puck frame. While you tighten the screws, work carefully so you do not let the wrench slip into the wires.
6. Test the puck by following any of the puck installation wizards in the Heat Capacity software.
7. Calibrate the puck. Refer to Chapter 5.

Figure 12.2.

Connecting the loose end of the broken wire to the pin in this manner is recommended over attempting to reattach the wire directly to the platform. It is very difficult to solder the wire in place and ensure the alignment of the platform when it is subsequently inserted into the sample-mounting station. Moreover, most solders, like SN96, have a superconducting transition at about 3.5 K. There is an anomaly in the heat capacity associated with this transition that will appear as part of the addenda. If only a single wire is repaired with an absolute minimum of solder, it is possible to restrict the size of this bump in the heat capacity to about 1% of the addenda heat capacity at this temperature.

For the adventurous, complete the following steps to reattach the loose end of the wire to the sample platform:

1. Remove the two screws that attach the wire guard to the puck frame, and then remove the wire guard. Save the screws.
2. Remove the two screws located on the bottom of the puck frame, and then remove the puck frame by carefully unplugging it from the eight sockets used for electrical contact. Save the screws.
3. Hold the puck frame so that it is upside down and then place it on a flat surface below a microscope or magnifying glass.
4. Using tweezers and a soldering iron, lightly touch the tinned tip of the iron to bond the wire to the pad. The amount of solder left on the pad should be an absolute minimum due to the anomalous heat capacity contribution to the addenda. The wire wets easily, so very little solder should be necessary to achieve a strong joint.
5. Use the screws to reattach the frame to the body of the puck.
6. Test the alignment of the sample platform by inserting it into the sample-mounting station. The platform is properly aligned if it is pulled snugly onto the platform holder when the vacuum pump is on and the puck interlock arm is engaged. If the platform is properly aligned, go to step 8.
7. If the sample platform does not fit squarely into the platform holder, the length of the repaired wire may have been altered during the repair. To fix this problem, simply touch the soldering iron to the pin (or for certain models, solder pad) to which the repaired wire is attached. While you touch the soldering iron to the pin, you should leave the mounting-station pump running and the puck interlock arm engaged. After touching the iron to the appropriate pin, the stress in the wire will be relieved, thus allowing the platform to slip into the proper position.
8. Place the wire guard on top of the puck frame. Locate the two screws you removed in step 1. Use the screws to attach the wire guard to the puck frame. While you tighten the screws, work carefully so you do not let the wrench slip into the wires.
9. Test the puck by following any of the puck installation wizards in the Heat Capacity software.
10. Calibrate the puck. Refer to Chapter 5.

12.5 Temperatures Read by User Thermometer on Standard PPMS and System Thermometer Do Not Match

If the puck thermometer temperature, as displayed in parentheses on the Model 6000 front panel during a heat capacity measurement, does not match the system thermometer temperature, there may or may not be a problem with the calorimeter puck. The Heat Capacity system operates in vacuum, so a small difference—usually less than 1%—between the puck temperature and the system temperature is likely; this temperature difference is the reason why the puck has its own thermometer. To maintain some moderate thermal contact between the puck and the bottom of the sample chamber, it is important that grease be applied to the puck fingers and that the fingers be periodically spread outward, as described in Section 12.2. To test the calibration of the puck thermometer and the platform thermometer, you complete the following procedure:

1. Perform the **Prepare Addenda Measurement** wizard from the **Installation Wizards** tab in the Heat Capacity control center in order to install the puck and purge the sample chamber as in preparation for measuring addenda.
2. Follow the steps in the wizard until you reach the **Puck Test Results** panel.
3. To verify the calibration at a specific temperature, simply set the new temperature manually from the front panel of the Model 6000 and wait until the temperature is stable.
4. Once the temperature is stable, press the **Test Again** button to compare the **Measured** and **Expected** values for the thermometers.

If the temperature difference is greater than 0.5%, the calibration of the puck thermometer is probably in error.

If, after performing the preceding test, the calibration is found to be bad, you may need to follow the puck calibration procedures in Chapter 5.

If you find that the platform or the puck thermometer is off more than about 1%, you may have a damaged puck. In this case, calibrating it again may help only temporarily or not at all. Please contact your Quantum Design representative if you need additional assistance.

12.6 Sample-Mounting Vacuum Pump Produces Excess Noise

After perhaps 200 hours of operation, the vacuum pump that supplies vacuum to the sample-mounting station may begin to produce a loud noise. Also, the suction of the pump may be reduced. This noise could indicate that the pump's diaphragm is broken. Contact Quantum Design to obtain a replacement diaphragm.

Troubleshooting Heat Capacity Measurements

13.1 Introduction

This chapter contains the following information:

- Section 13.2 discusses indicators of measurement quality in the data file.
- Section 13.3 discusses some common errors that are made during heat capacity measurements.

13.2 Indicators of Measurement Quality in the Data File

When viewing the measurement data contained in a data file, it is important to know whether the data is good or not. Several fields in the data file can be viewed to verify data integrity. While the following sections suggest signs of bad data, the sections do not always give a remedy. Usually the problem can be traced to a sample that is too small or poorly attached to the sample platform. Using nonrecommended values for measured parameters could also be the source of problems.

As a general suggestion, all of the following fields, and many others, can be examined by plotting the field versus Sample Temperature. Often, poor quality measurements are limited to a fraction of the full temperature range of a series of measurements. Sudden changes in a data value with temperature should be investigated carefully.

13.2.1 Heat Capacity Error

If the data file contains data for a sample, you should look at both Samp HC and Samp HC Err. Samp HC Err gives the estimated error contained in the sample heat capacity as given by Samp HC. Samp HC Err corresponds to the error bars. This error is determined by the fitting routine and the quality of the fit as discussed in Chapter 4. If Samp HC Err is more than approximately 10% of Samp HC, you may have a problem.

For an addenda measurement, you should check Addenda HC Err as compared to Addenda HC.

13.2.2 Fit Deviation

The Fit Deviation field contains the normalized chi square for the fit. Where the fit deviation is dominated by thermometer noise this value is approximately 1. Generally, smaller values are better. For most measurements below 200 K, this value is approximately 10. For measurements above 200 K, this value could be as large as 100. If the value is substantially larger than this, the automatic fitting routine may have failed to produce a correct fit, or a glitch occurred during the measurement. The HC Err as described in Section 13.2.1 should reflect this bad fit. Remove the bad data point from the data set if it appears that a spurious glitch occurred.

13.2.3 Sample Coupling

If the Fit Deviation is not too large, the Sample Coupling field indicates how well or how poorly the sample is thermally attached to the sample platform. 100% indicates perfect coupling. The exception is if the fitting algorithm failed to converge to a two-tau fit (see Section 4.3). It is important to distinguish between perfect coupling and a failed fit. For a sample measurement, a failed fit is indicated by the Time Const (tau2) value equal to zero.

If Sample Coupling is less than approximately 90% or the two-tau fit has failed, you might be advised to try to remount your sample and then re-measure it.

Sometimes, certain samples exhibit a large differential thermal contraction with respect to the sapphire calorimeter platform. A dramatic drop in the Sample Coupling (or a failure of the two-tau fit) as the temperature is reduced may indicate a strain between the sample and the platform that results in cracked grease. This is usually only a problem for samples with a large flat surface contacting the calorimeter platform, but can also be a problem for samples with structural transitions. Better result can sometime be obtained by breaking the sample into a number of smaller pieces that are individually attached to the grease on the platform. The theory is that even with the same relative differential strain, the absolute strain will be smaller for each of the pieces, reducing the chance of cracking the grease when the temperature is lowered.

The 90%-rule for Sample Coupling may need to be ignored at temperatures below about 1 K. The reason is that thermal coupling between the sample and the platform may be intrinsically poor at these temperatures (see Section 9.2.1). At these temperatures, a sample coupling of even 70% may be considered acceptable if the fits look reasonably good.

13.2.4 Other Fields to Examine

When you run a heat capacity measurement, you specify a temperature rise in either kelvin or percent. When examining the resulting data, verify that the Temp Rise field is roughly the same as that specified by the measurement parameters. That is, if you specified a Temp Rise of 2% and the Sample Temp field is 6, Temp Rise should equal about 0.12, which is 2% of 6. If it is off by more than approximately 50%, this may be a problem. If the temperature rise is smaller than requested, there may be a problem with the vacuum. This can also be confirmed by examining the Pressure or the Wire Conductance data items.

The Pressure field indicates the pressure at the top of the sample chamber. Normally this will be less than about 0.001 torr. A value larger than this could indicate a vacuum leak or the possible failure of the high vacuum system. Heat capacity measurement acquired under these circumstances should be considered suspect. At temperatures below about 10 K, the Pressure will be of limited diagnostic utility. For example, the charcoal (see Figure 3-8) would absorb Helium leaking into the cold end of

the sample chamber at 2 K. This gas would not be seen by the pressure gauge but would still affect the heat capacity measurement. (See section 13.3.1).

Wire Conductance is computed by the fitting algorithm for both addenda and sample measurements. This field can be a valuable diagnostic for determining the effective gas pressure at the calorimeter. It is especially useful below about 10 K where the pressure gauge may not be sensitive to leaks because of the charcoal. By plotting the Wire Conductance vs Temperature you should verify that the Wire Conductance always increases monotonically with temperature. Also, there should not be significant changes in the Wire Conductance as compared to previous measurements, including the values in the Wire Conductance table stored in the `.cal` file.

When you run a heat capacity measurement, you also specify the measurement time in units of time constants. For example, if you specified 1 for this, the Meas Time field should be approximately equal to the Time Const tau 1 field. If you specified 2, Meas Time should be approximately 2 times larger. Deviations of more than approximately 50% could indicate a problem.

13.3 Common Mistakes and Interesting Results

This section describes common experimental errors associated with performing heat capacity measurements as well as some subtle artifacts specific to the Quantum Design Heat Capacity system. The following list is concerned primarily with effects that not specifically discussed in other sections of this manual. For an updated list of known results, please refer to the Quantum Design website at www.qdusa.com.

13.3.1 Helium Condensation/Adsorption on the Sample or Platform

At temperatures below about 6 K, it is possible to see an anomalous heat capacity due to collection of helium on the platform and in the sample if the sample is particularly porous. This usually appears as a low broad peak around 3 K. The peak will often be worse if the temperature has been below 5 K for an extended period of time. Cycling the temperature up to 20 K will usually temporarily fix the problem (see section 7.5). The charcoal holder on the contact baffle assembly is meant to fix this problem. However, in some cases it can become prematurely saturated. The problem is most likely to appear if the charcoal holder was inadvertently left off or you are measuring a sample that has a greater affinity for helium gas than charcoal.

At DR temperatures, helium contamination can result in superfluid films with dramatic effects including thermal short circuits. See Section 9.2.5 for more information.

13.3.2 Air Leaks and Improper Purging

Another effect related to the helium condensation problem is that of ice contamination of the sample. It is important to purge the sample chamber at temperatures above about 280 K. Even at room temperature, the neck of the PPMS sample chamber can remain cold enough to freeze water and some air constituents. Unless the sample chamber is periodically baked-out at higher temperatures, such that the neck is warmed, there will be a gradual collection of ice on the walls of the sample chamber. If you purge the sample chamber while the puck is cold, the helium gas can eject ice from the walls of the

sample chamber and deposit it on the heat capacity platform. The Heat Capacity system is sensitive enough to detect such contamination. Experimentally, the magnitude of the problem can be seen by repeatedly purging and performing an addenda measurement below, for example, 50 K. With each subsequent purge, the addenda heat capacity will increase slightly. You may even see peaks resulting from an antiferromagnetic transition in frozen oxygen. In rare cases, the air ice may result from a leak in the sample chamber. However, this is usually just a result of poor sample chamber hygiene.

13.3.3 Non-smooth Specific Heat Between 200 K and 300 K due to Anomalous Specific Heat of Apiezon N Grease

Apiezon N Grease exhibits a slight anomaly in its specific heat near its melting point between 260 K and 325 K. This can result in non-smooth results in this temperature range. Please see section 6.2.4.1 and *Quantum Design Heat Capacity Application Note 1085-152A* at www.qdusa.com for more information and specific recommendations.

13.3.4 Scatter in Heat Capacity Data Below 2 K Due to Vibration of Heat Capacity Platform

Below about 2 K, vibrational warming of the heat capacity platform can cause increased scatter in the heat capacity data when using both the Helium-3 insert and the DR insert. Helium-3 systems with vertical sample stages (Section 8.2.3) appear to be the most susceptible to this effect. The platform stabilizer plug (Figure 8-1) can be used to suppress this effect.

With both the Helium-3 insert and the DR insert, some modest vibration isolation of the cryostat from the floor can substantially reduce this effect. Other sources of vibrations come from the PPMS insert itself. This includes oscillations in the gas-flow system and also the turbo pump high-vacuum system (for systems without a cryopump).

A related effect occurs when measuring in a magnetic field where a voltage is induced on the thermometer readback circuit when the calorimeter platform oscillates in the magnetic field. This effect can be identified by an oscillator pattern in the temperature versus time trace in the measurement status viewer.

Contact Quantum Design for help if you are unable to eliminate spurious effects from the vibration sources.

13.3.5 Steps in Heat Capacity vs Temperature

The Heat Capacity control electronics use a set of fixed excitation currents applied to the platform thermometer depending on the temperature. As described in section 5.5, the measured resistance of the platform thermometer can depend slightly on the excitation current because of self-heating effects, especially at lower temperatures. This is why there are different thermometer calibration tables for each excitation current used by the controller. The calibration procedure has been designed to condition these tables to minimize the appearance of a transition in the heat capacity data at temperatures where the excitation current is changed. However, under some circumstances, these steps in the resulting heat capacity vs temperature are still visible. One way to distinguish between these steps which are artifacts of the calibration, and real heat capacity steps, is to examine the Time Const (τ) field versus Sample Temperature. A real step in the heat capacity would also appear in the Time

Const (tau1) field. If the step was purely due to the calibration, it would appear in the Heat Capacity (or Addenda HC) but not in the Time Const field.

Sometimes these calibration steps result from a flawed calibration run and can be fixed by rerunning the calibration procedure. Also, these steps may result from a short to ground someplace in the calorimeter wiring (probe wiring or on the puck itself), where self-heating is enhanced by unbalancing the impedance of the drive circuit.

13.3.6 Loose Radiation Shield on Standard Puck Causing Elevated and Inconsistent Sample Temperatures

If the lid on the standard calorimeter puck is too loosely attached, and it is cooled down under high-vacuum conditions, the temperature of the lid can lag the temperature of puck by possibly hours and tens of kelvin. Then, thermal radiation from a warmer lid will cause the calorimeter platform (and hence Sample Temperature) to be warmed compared to the System Temperature. Heat capacity measurements acquired under these conditions will occur at higher temperatures than requested, possibly by several kelvin. The amount of heating offset from this effect will become smaller as the lid temperature comes to equilibrium with the puck. When acquiring heat capacity measurements versus temperature, this may appear as an elevated and inconsistent spacing of Sample Temperatures reported in the data file.

The most effective solution to this problem is to make sure that the radiation shield (lid) fits tightly onto the puck frame. See section 3.2.1.2 for information on making the lid fit tightly.

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