



Heat Capacity Option for the PPMS

5 September, 2002

Heat Capacity Summary

- What is heat capacity?
- Why do customers want it?
- Measurement techniques
- QD implementation
- How to calibrate and measure
- Common problems

What is Heat Capacity?

- The amount of heat required to raise the temperature a given amount:

$$C = dQ/dT \approx \Delta Q/\Delta T$$

- Q is heat in energy units (Joules)
- Thermal analog to mass
 - Mass describes how hard it is to change an object's velocity
 - Heat capacity describes how hard it is to change an object's temperature

Heat Capacity vs. Specific Heat

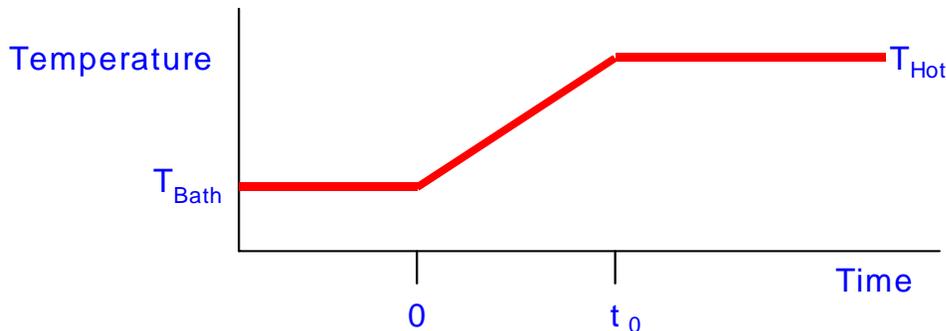
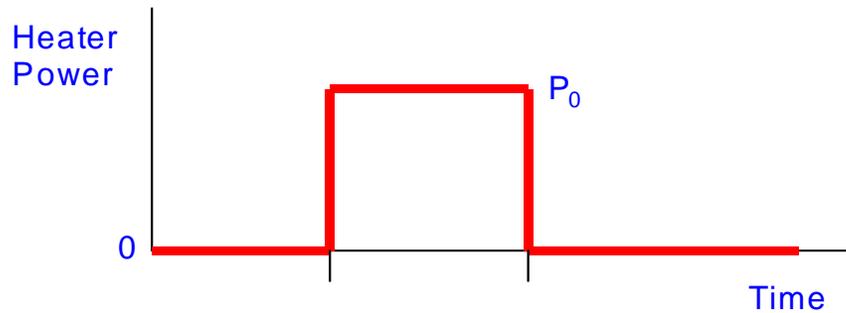
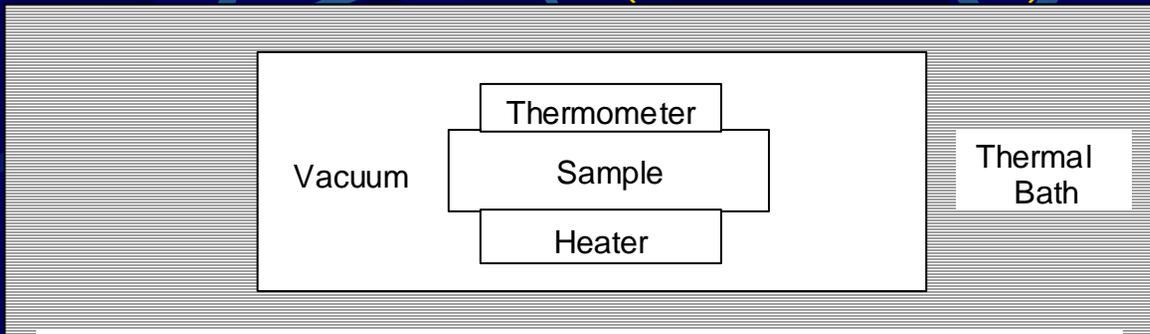
- Heat capacity scales with the amount of material
 - Common units: J/K, $\mu\text{J}/\text{K}$, cal/K
 - Extrinsic: depends on sample size
- Specific heat =
(Heat Capacity)/(amount of material)
 - Common units: J/g-K, $\mu\text{J}/\text{mg-K}$, J/mol-K, cal/mol-K
 - Intrinsic: does not depend on sample size

Why do customers want it?

- Very useful measurement for many fields of physics and chemistry. Examples:
 - Lattice properties
 - Phase transitions
 - Schottky anomaly
- Traditionally very difficult to measure heat capacity
 - Accessible to specialists only
 - QD automated system makes it available to much wider group

Adiabatic Measurement Technique

(Not what we use)



$$\Delta T = T_{\text{Hot}} - T_{\text{Bath}}$$

$$\Delta Q = P_0 \cdot t_0$$

$$C = \Delta Q / \Delta T$$

Require C constant
in temp. range ΔT

Adiabatic Technique

● Advantages

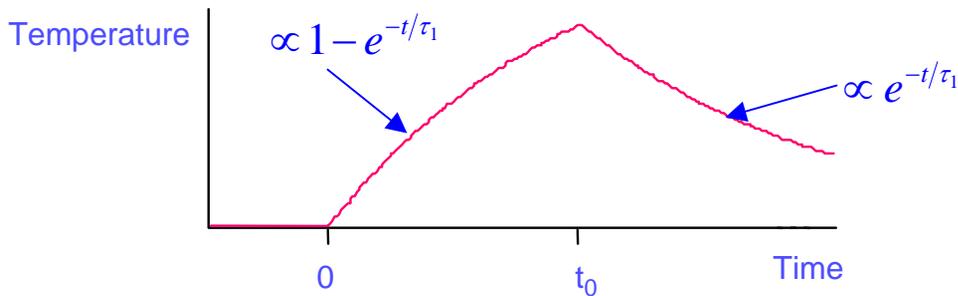
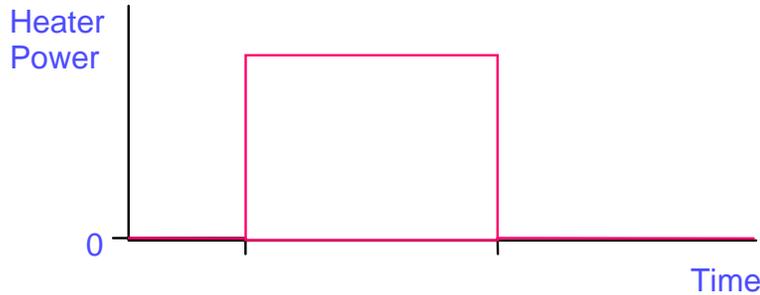
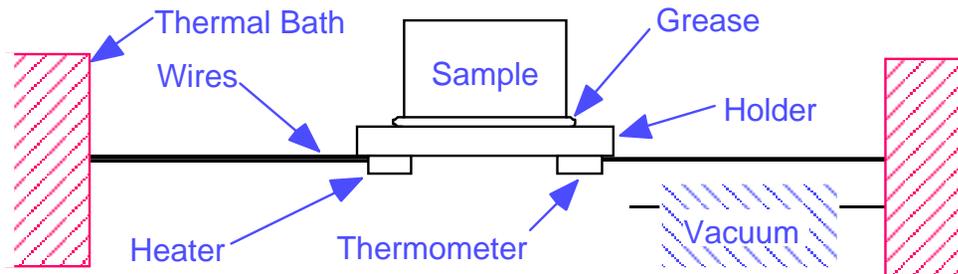
- Very good accuracy
- Easy to interpret results
- Can measure first order transitions

● Disadvantages

- Need extremely good thermal isolation
- Only works with big samples
- Tedious sample mounting
- Must correct for radiation loss at high temperature

Relaxation Technique

(Simple Model)



$$\tau_1 = C/K$$

K = thermal conductance of wires

Require C constant in temp. range ΔT

Relaxation Technique

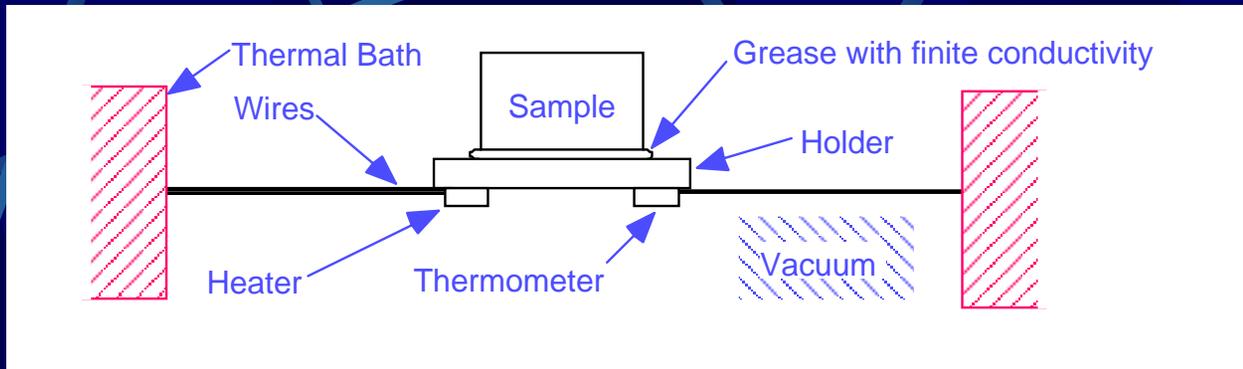
● Advantages

- Can measure small samples
- Easy sample mounting
- Good accuracy

● Disadvantages

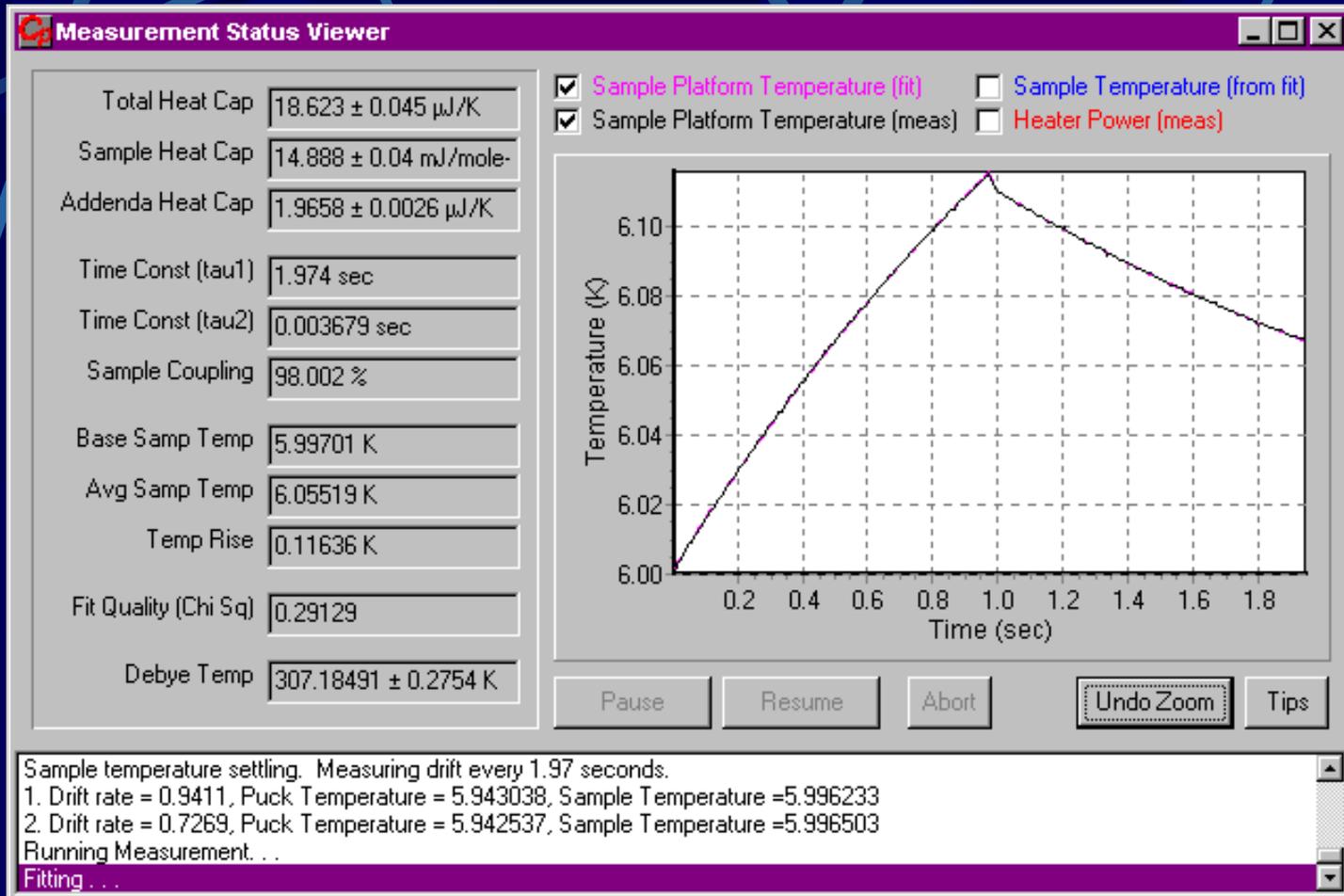
- Complicated curve fitting
- Slow for big samples
- Results can be difficult to interpret if conditions are not ideal

Relaxation 2-tau model



- Two time constants
 - τ_1 : time for equilibrium of chip and sample with thermal bath
 - τ_2 : time for equilibrium of sample with chip
- Sample coupling: measure of quality of sample mounting
 - Should be $> 90\%$ for good measurements

2-tau Relaxation Curve



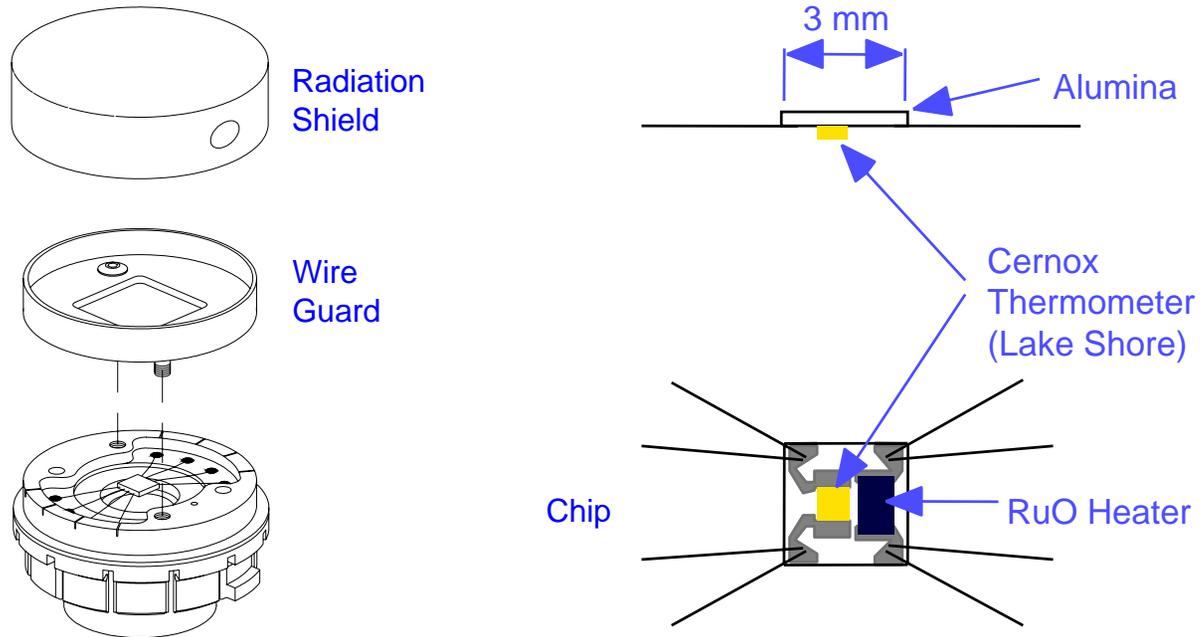
QD Heat Capacity Option Features

- Standard heat capacity
 - Measure from 1.9 K to 400 K
- He-3 heat capacity
 - Measure from 0.4 K to 350 K
- Fully automated measurement sequencing and curve fitting
- Automatic addenda heat capacity subtraction

Components of QD Heat Capacity Option

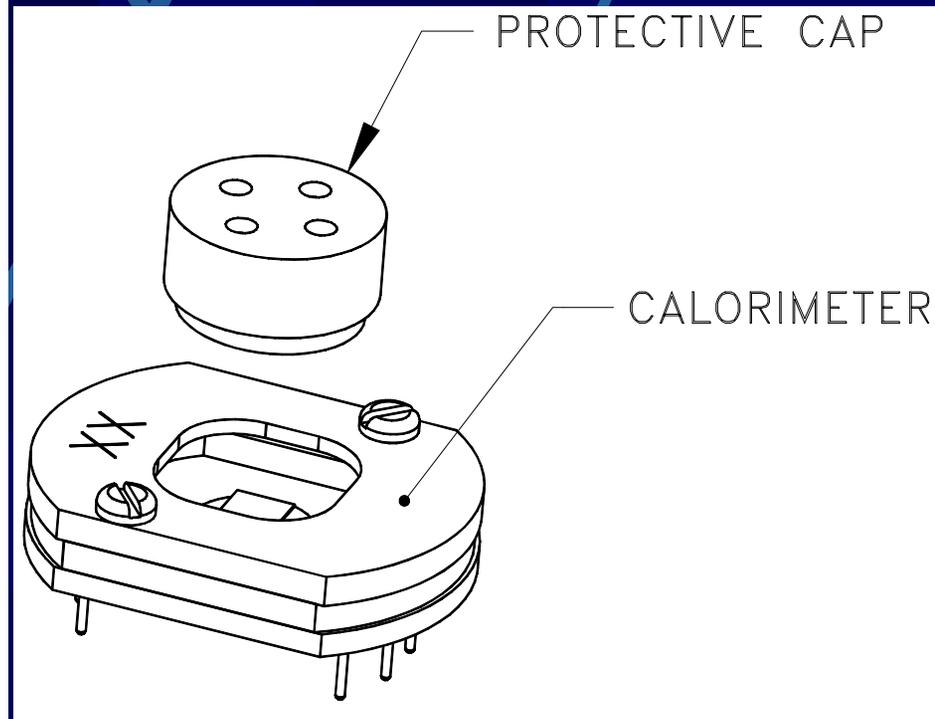
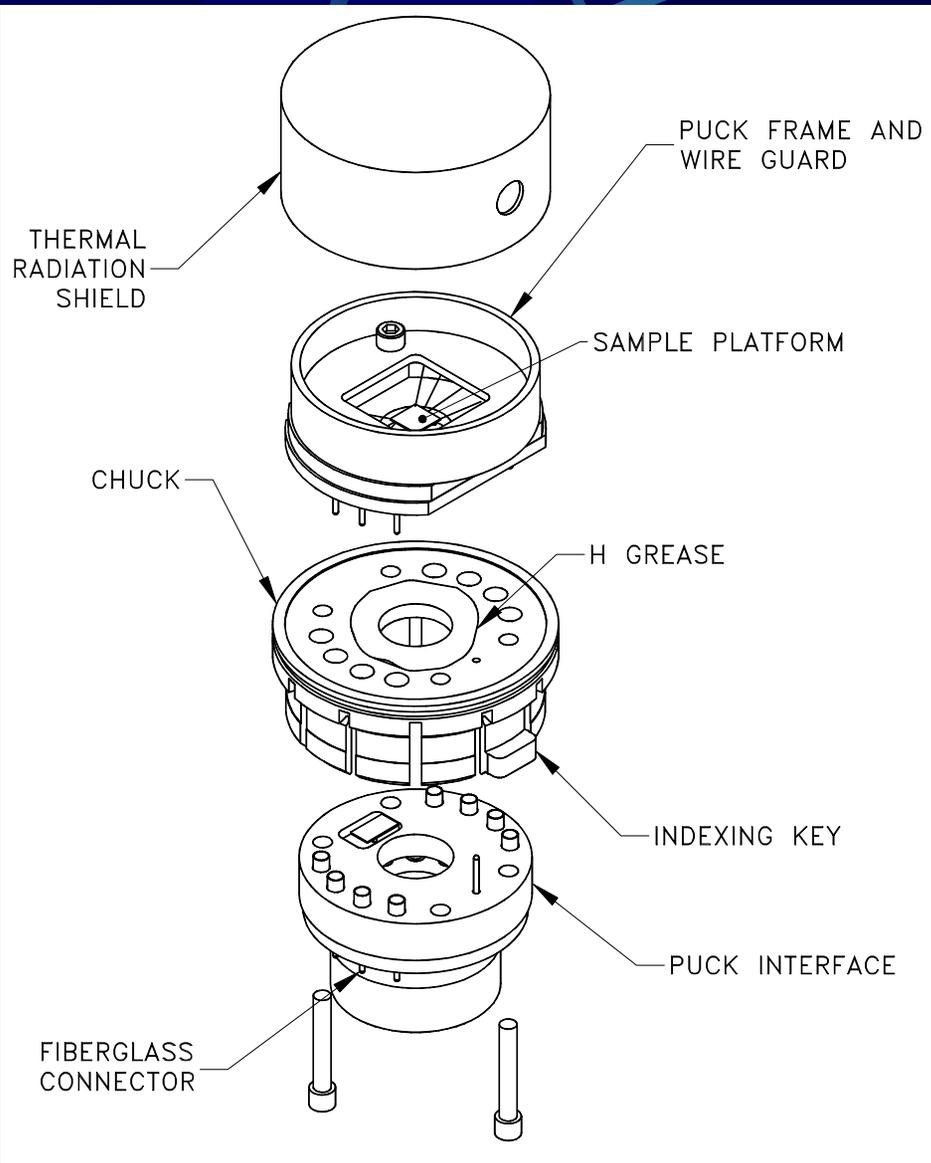
- Standard heat capacity puck or He-3 heat capacity frame
- HC DSP card in option crate
- HC Cable
- High vacuum (includes contact baffle with charcoal)
- Sample mounting station
- HC Software
- PPMS

Standard HC Puck



Thermometer in puck for UserTemp control

Std Puck vs. He-3 Frame



Std Puck vs. He-3 Frame

	Std Puck	He-3 Frame
Number of wires	8	4
Wire size	3 mil	2 mil
Chip thermometer	1050 Cernox	1020 Cernox
Puck thermometer	1050 Cernox	None

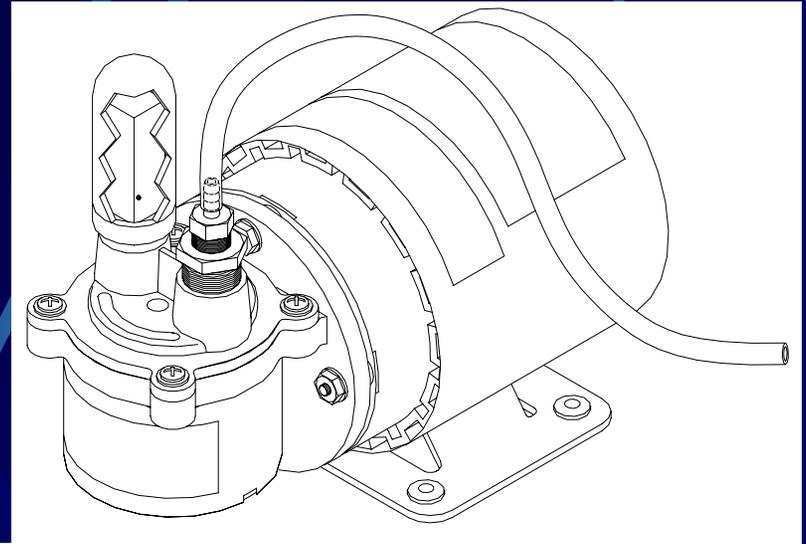
HC Pucks Can Be Tested With User Bridge

Puck connections when using User Bridge Cable

Component	User Bridge Channel
Chip Heater	1
Chip Thermometer	2
Puck Thermometer (std) He-3 Thermometer (He-3)	3

Sample Mounting Station

- Holds chip in place to allow application of grease and mounting and dismounting of samples

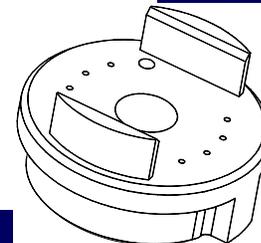
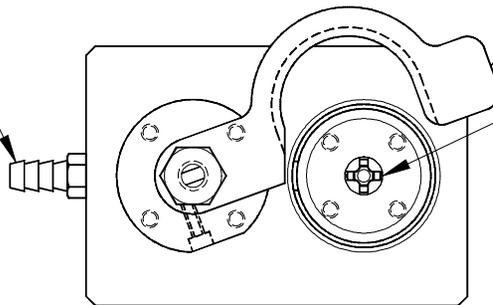


HOSE BARB FOR
VACUUM HOSE

PUCK INTERLOCK ARM

PLATFORM HOLDER

SAMPLE MOUNTING
STATION ADAPTER



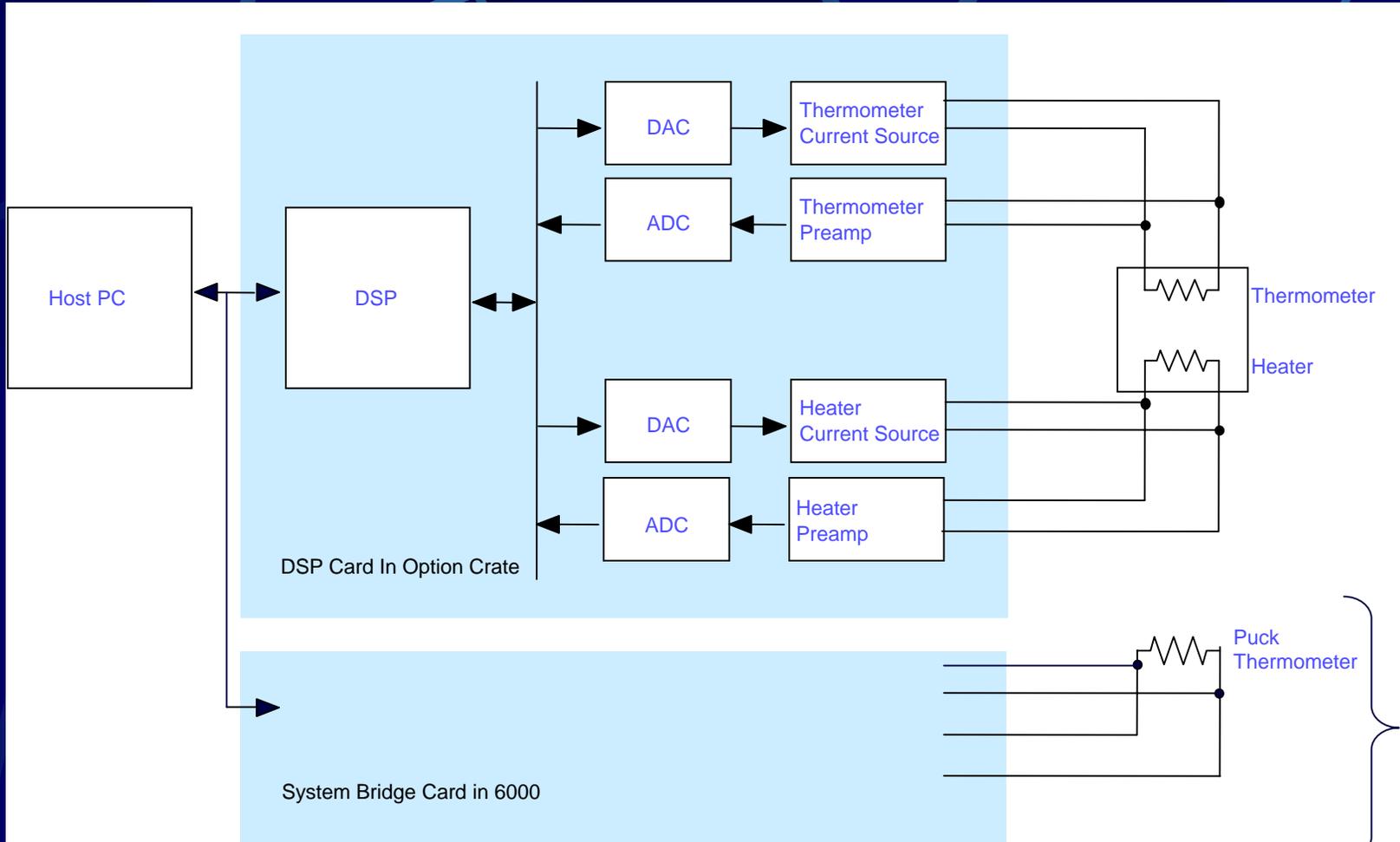
HC DSP Card

- Current source for heater
 - Voltage readback for heater
 - Current source for chip thermometer
 - Voltage readback for chip thermometer
 - DSP to control everything and store data
 - Lives in Option Crate Slot 3
- } 4-probe power measurement
- } 4-probe resistance measurement

DSP Card Calibration File

- HC software version 2.5.5 or older
 - HeatCap.hwc
 - No entry in HeatCap.ini
- HC software version 2.5.6 or newer
 - HeatCap-d-###.hwc
 - Entry in HeatCap.ini:
SerialNumber=d-185
 - Problem: don't delete HeatCap-d-###.hwc if serial number line is present in ini file.

Block Diagram



Replace with He-3
thermometer and user
bridge for use with he-3

HC Software

- Calibration

- Automated puck calibration performed by final test and by users

- Heat capacity measurement

- Sets up DSP
- Reads back heater power and thermometer resistance vs. time data from DSP
- Converts therm. R to T using cal file
- Fits data to calculate heat capacity

Puck Calibration Pass 1

- Calibrate therm. R vs. T
 - Cal puck and chip therm. for std puck
 - Cal chip therm. only for he-3 frame
 - PPMS block or he-3 therm. is reference
- Want exchange gas for good calibration
 - Purged (NOT high vac) chamber state
 - Remove charcoal from contact baffle
- He-3 must operate in hi vac below 20 K
 - Shorting fixture to insure accurate calibration
- Change .pkd file to change calibration temperatures

Puck Calibration Pass 2

- Performs addenda heat capacity measurement – heater addenda table
 - Need hi vac and charcoal on contact baffle
 - Need to remove shorting fixture for he-3
 - Use sample mounting station
 - Be careful!!
- Measures wire thermal conductance
 - Used later to predict P needed to get desired ΔT
- Measures heater resistance
 - Used later to predict I needed to get desired P

HC Measurement

- Put puck in sample mounting station and apply grease to chip
 - We supply Apiezon N grease for $T < 295$ K and H grease for higher T.
- Measure addenda heat capacity
 - Addenda table stored in puck cal file
- Mount sample using sample mounting station
- Measure sample heat capacity
 - Software automatically subtracts addenda heat capacity by interpolating in addenda table

MR Calibration

- Performed by user, not QD!
- Requires MR calibrated chamber as reference for std puck
- He-3 therm is reference for he-3 frames
- Calibrate R vs. T at discrete fields
 - NOT a 2D table!
 - User should calibrate at every field he wishes to use for measurement

Click on the links below to
view video files

- Applying the grease
- Mounting the sample

Common Problems

- DSP errors
- Bad wire conductance data during Pass 2
- Don't get desired temperature rise
- Poor sample coupling
- Addenda heat capacity depends on field

DSP Errors

- Make sure you are using most current software revision
- Reboot everything and try again
- Failure report so we can fix the problem!

Bad Wire Conductance Data During Pass 2



- Usually caused by poor high vacuum
- Improve vacuum and rerun pass 2

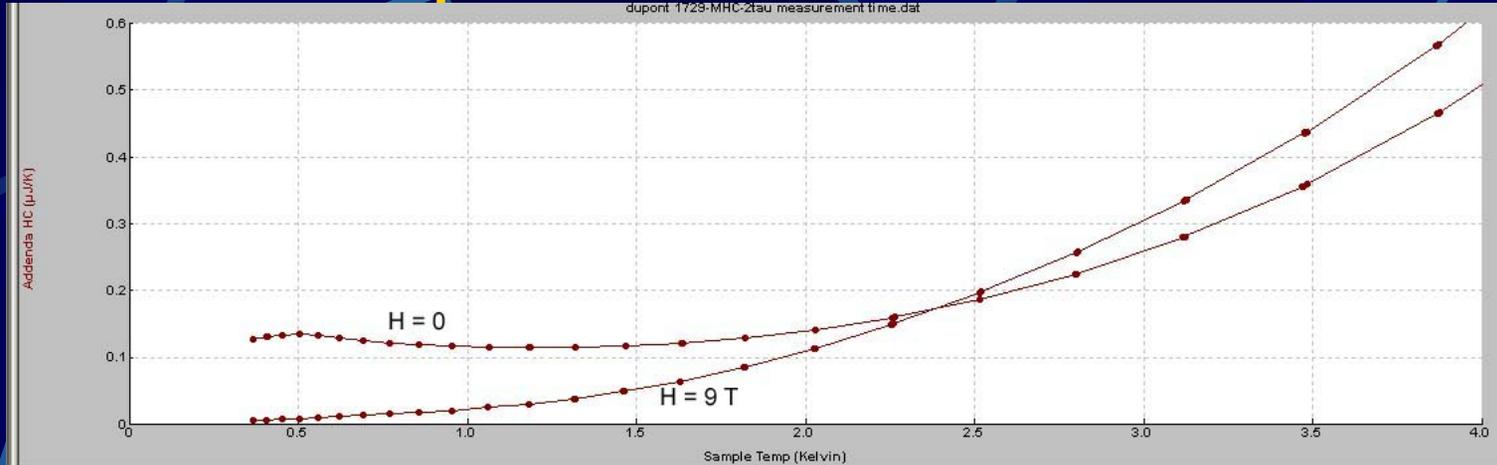
Don't get desired temperature rise during measurement

- Poor high vacuum during measurement
 - Will have bad wire conductance data in measurement data file
 - Improve vacuum and run again
- Bad wire conductance data in calibration file
 - Probably due to poor hivac during pass 2
 - Fix and rerun pass 2

Poor Sample Coupling

- Less than 90% is considered poor
 - Can get decent data with lower sample coupling
- Improve sample mounting to fix it
 - Use flat samples to maximize contact area
 - For samples with large thermal contraction, float on grease (don't press down hard)
 - Break up large samples to allow for thermal contraction
 - Try a different grease (ex: Wakefield 120)

Heat Capacity of Addenda Depends on Field



- Due to impurities chip materials
- Measure addenda in field before measuring heat capacity
- Use correct addenda table for each field
- We are working on chip without field dependence

Heat Capacity Quiz

(Answers in Red)

1. High vacuum is required for which of the following heat capacity functions (standard heat capacity, not he-3)? (Circle all that apply)

(a) Addenda Heat Capacity Measurement

(b) Sample heat capacity measurement

(c) Calibration Pass 1

(d) Calibration Pass 2

2. How is the sample heated in a heat capacity measurement? (Circle one answer)

(a) By heating the sample chamber

(b) By applying current to a resistor on the chip

(c) Thermal radiation

(d) By applying current to the sample

3. Why is extremely noise-free temperature control required for good heat capacity measurements?

The temperature rise during a heat capacity measurement is typically only 2 % of the temperature. If the temperature noise were comparable to 2 %, then it would be impossible to obtain quiet temperature vs. time data to fit with the relaxation model and extract the heat capacity. The temperature noise must be small compared to the temperature rise.

4. The DSP card has failed on a customer's system. What do you need to send him in order to correct the problem?

Send a new DSP card, the corresponding HeatCap-d-###.hwc file. You also need to send a new HeatCap.ini file which points to the new .hwc file, or change the existing .ini file to point to the new .hwc file.

5. A customer sees an unexpected peak in his heat capacity data around 273 K. What is the most likely cause of the problem?

Water freezes and melts at 273 K. The heat capacity of water has a peak at this phase transition. Therefore, the most likely cause of the unexpected peak is water on the heat capacity chip.

Water could get on the chip in a number of ways. A leak in the high vacuum system could result in water in the sample chamber. Also, water ice often freezes on the sample chamber walls when the chamber is open. Pieces of ice can then fall on the heat capacity chip during a purge and seal operation.

6. (Extra credit-difficult). A sample has a phase transition with a latent heat which causes an extremely high and narrow peak in the heat capacity at the phase transition. What do you expect to happen if a temperature vs. time trace during a heat capacity measurement crosses the phase transition? Draw the expected sample temperature vs. time for a relaxation measurement. Do you expect the heat capacity software to yield accurate results for this case?

When the heat capacity has a large peak, it takes a lot of heat to raise the temperature of the sample a very small amount. Therefore, the temperature will rise very slowly at the transition temperature if the power input is constant as in a heat capacity measurement (see graph). The software model does not take account for such an effect, so the results from such a measurement are not accurate. (The figure is an exaggeration to illustrate the effect.)

