Quantum Design

PPMS Application Note 1085-153

Reducing Time to Calibrate Heat Capacity Pucks by Limiting the Temperature Range

Every heat capacity puck must be calibrated before use, and it takes about 20 hours to perform each standard calibration over the entire temperature range. However, when appropriate for your work, you can reduce that time by limiting the temperature range used for the calibration. This note explains how to modify the calibration process.

Standard Calibration Process

The .pkd file is used to calibrate the puck in two passes, each taking about 10 hours. In pass one (Pass 1), the MultiVu calibration procedure measures and records the temperature-dependent resistance of the platform and puck thermometers. Pass two (Pass 2) is used to measure the temperature dependence of the platform-heater resistance and the thermal conductance of the connecting wires that suspend the platform. Respectively, these values are used during heat capacity measurements to predict the amount of current that is needed to produce the desired power and the amount of power that is needed to produce the desired ΔT. The calibration procedure stores the data in a separate Puckxxx.cal file for each puck.

Modified Calibration Process

For research that is performed at one or more specific temperature ranges, the .pkd file can be modified to calibrate the thermometers for a specific temperature range rather than for the entire range, thus reducing calibration time. This modification applies to Pass 1 of the .pkd file. The procedure for Pass 2 is then modified to calibrate the temperature dependence of the heater resistance

(R_M) and the wire conductance (K_M) at only one temperature (305 K). We supply an Excel spreadsheet (Figure 1) and procedure that uses the latter data to generate the temperature-dependence data for the desired range of temperature.

Figure 1. "Stdpuck heater res" worksheet in the 1Point_Pass 2_data.xls spreadsheet

The spreadsheet for Pass 2, 1Point_Pass2_Data.xls, contains tables of temperature-dependence data [see column 2, R_T (Ohms)] derived from several calibrated Puckxxx.cal files. The spreadsheet procedure combines the derived data with the temperature-dependence of the heater resistance (R_M) or the wire conductance (K_M) at 305 K, using the resistance (or conductance) ratio shown in the formula below.

$$R_{Cn} = \frac{R_M}{R_T} R_{Tn}$$

Definitions

R_M & K_M: The resistance/conductance measured at 305 K during the calibration process; it is recorded in the new `Puckxxx.cal` file

R_T & K_T: The table resistance/conductance at 305 K

R_{Tn} & K_{Tn}: The nth table resistance/conductance

R_{Cn} & K_{Cn}: The nth calculated resistance or conductance for the puck being calibrated. This value will be used for Pass 2 data for the `Puckxxx.cal` file.

Note: The accuracy of R_{Cn} and K_{Cn} is not critical to the measured heat capacity. These parameters are used to set the heater current to the level that will achieve the desired ΔT during heat capacity measurements. Hence, the accuracy of the measured heat capacity will not be affected by small inaccuracies in R_{Cn} and K_{Cn}.

1Point_Pass 2_Data.xls Spreadsheet

The `1Point_Pass2_Data.xls` spreadsheet has six worksheets. The first two, "Stdpuck heater res" (Figure 1) and "Stdpuck wire cond," generate Pass 2 data for a standard puck when the resistance (R_M) or conductance (K_M) measurement is entered into the formula. The next two worksheets, "He3Puck heater res" and "He3Puck wire cond," are used in the same way to generate Pass 2 data for a Helium3 puck. The generated temperature-dependence data are automatically entered into one of the other two worksheets, "new_stdcal_data" or "new_He3cal_data." You will copy this data to the `Puckxxx.cal` file.

The following section explains how to do the modifications.

Important: Save any modified `.pkd` files with a new file name so that you do not overwrite the original files.

PROCEDURES

Updating the HeatCap.ini File

1. Open HeatCap.ini located under
    \\QdPpms\HeatCapacity\System.

    In the General section of the file, add the following command,
    "`EnableChamberCommands=0`", as in Figure 2:
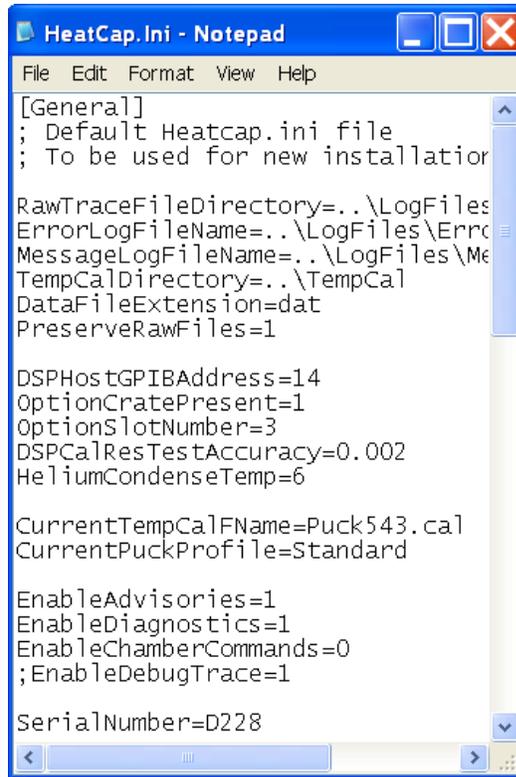
Figure 2. HeatCap.ini File

This command will disable the heat capacity software's ability to automatically control the chamber status. This command is required in order to perform the calibration for a limited temperature range. Therefore when executing the heat capacity calibration wizard, you must manually purge the chamber before beginning Pass1 and set the chamber to HiVac when performing Pass2. Once the calibration is complete, you can reactivate the automatic chamber control of the heat capacity software by changing the 0 to a 1.

`EnableChamberCommands=1`

2. Save the changes to the *.ini file. Be sure to reactivate the Heat Capacity option in order for the changes to be sent to the Model 6000.

Pass 1: Modifying Temperature Range

1. Using the `Notepad` program, open the `Standard.pkd` file (C:\QDPpms\HeatCapacity\TempCal\Standard.pkd) and **Save As** `1PointPass2.pkd` in the `HeatCapacity\TempCal` directory. (For the Helium3 Heat Capacity puck, open the
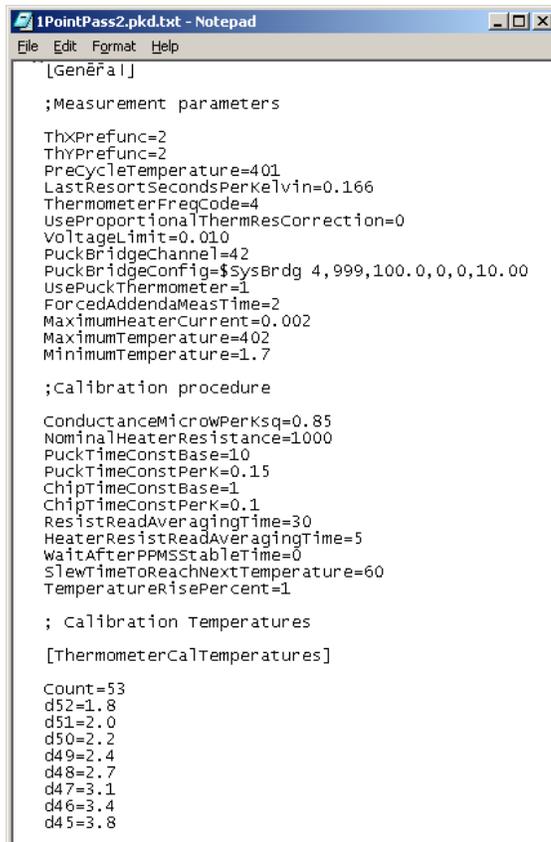
He3Puck.pkd file and **Save As**
1PointPass2He3.pkd.*)*

The 1PointPass2.pkd File

As shown in Figure 3, the file begins with a general
list of measurement parameters and calibration
procedures. Calibration temperatures begin with the
heading [ThermometerCalTemperatures], as shown
in the lower portion of the figure.

The thermometer-temperature section first lists the
total number of measurements (**Count**=53). Then it
lists the measurement numbers (e.g., d52) and the
temperatures to be used (e.g., d52 = 1.8).

Important: The **Count** runs down (from dN to d0).
Therefore, the total **Count** is N + 1.

Figure 3. Top section of the 1PointPass2.pkd file
(before the temperature range has been edited)

2. Scroll down to the section labeled
   [ThermometerCalTemperatures], shown in
   Figures 3 and 4, which lists the temperatures at
   which the thermometers will be calibrated.

```
[ThermometerCalTemperatures]

Count = 23
d22=2.0
d21=2.2
d20=2.4
.
.
.
d3=14.9
d2=16.5
d1=18.0
d0=20.0
```

Figure 4. The thermometer calibration section of an
edited 1PointPass2.pkd file.

3. Revise the list by deleting temperatures that are
   not part of your desired range.

4. Change the numbers (d##) and **Count** to reflect
   your changes. For example, based on the
   temperature list in Figure 4, there will be 23
   measurements, and the calibration procedure
   will measure the resistance of the
   thermometers from 2.0 K to 20.0 K.

5. **Save** the file but leave it open.

Next, you will change the section of the file that
controls Pass 2 measurements.

Restricting Temperature for Pass 2

1. Scroll down to the [HeaterCalTemperatures]
   section of 1PointPass2.pkd and change the
   list as shown in Figure 5, so that only one
   measurement will be taken (at 305 K).

```
[HeaterCalTemperatures]

Count=1
d0=305
```

Figure 5. Heater calibration temperature section of
an edited 1PointPass2.pkd file

The calibration procedure will use this list to
measure the heater resistance and the wire
conductance at 305 K. (The data for the rest of the
temperature range will be generated from the
1Point_Pass2_Data.xls spreadsheet.)

2. **Save** your changes and close the file.

CAUTION!

Do not overwrite the Standard.pkd file.

3. Run the calibration procedure described in Section 5.3, "Calibrating a Puck," of the *PPMS Heat Capacity Option User's Manual*. When you are asked to enter the type of puck to calibrate, select 1PointPass2.

Note: *The automatic chamber operations performed by the heat capacity software were disabled when performing the procedures above, "Updating HeatCap.ini File". Be sure to purge the chamber before starting Pass1 of he calibration process. Good thermal exchange is required inside the chamber for Pass1 in order to properly calibrate the thermometers. Therefore do not set HiVac for Pass1. For Pass2, be sure to set HiVac before starting the calibration.*

The data for the puck that is being calibrated will be recorded in a new file named Puckxxx.cal, which will be located in the C:\QDPpms\HeatCapacity\TempCal directory.

Generating the Data

Once the wizard has completed the calibration, use Excel to open the 1Point_Pass2_Data.xls spreadsheet and use Notepad to open the new Puckxxx.cal file (from Step 3 of "Restricting Temperature"). You will copy the relevant data from Puckxxx.cal (Figure 6) to 1Point_Pass2_Data.xls, as explained below.

[Temp_HtrRes]

XFuncCode=1
YFuncCode=0
XName=Temp
YName=HtrRes
Count=1
306.15466,1291.491 (R_M *at ~306 K for the heater*)
.
[Temp_Cond]
XFuncCode=2
YFuncCode=2
XName=Temp
YName=Conductance
Count=1
306.15466,0.00027950458 (*K_M at ~306 K for wire conductance*)

Figure 6. A portion of the Puckxxx.cal file, showing the measured resistance and conductance data. Commas in the readings separate the temperature (306.15466) from the R_M (1291.491) and the K_M (0.00027950458), here underlined for emphasis. You copy these values to the Excel worksheets, "stdpuck heater res" and "stdpuck wire cond" in the 1Point_Pass 2_Data.xls spreadsheet.

1. 1Point_Pass2_Data.xls spreadsheet

   • Activate the "Stdpuck heater res" worksheet

   • Leave 1Point_Pass2_Data.xls open

2. Puckxxx.cal file

   • Scroll down to the [Temp_HtrRes] section.

   • **Copy** the measured resistance (R_M) for the heater; remember to select only the numbers after the comma, as shown in Figure 5.

   • Leave Puckxxx.cal open.

3. 1Point_Pass2_Data.xls "Stdpuck heater res" worksheet (Helium3 option: "He3Puck heater res" worksheet)

   • **Paste** R_M into cell B2.

   As each value is pasted into cell B2, you will see the third column [KC (W/K) or RC (Ohms)] of that sheet fill with values generated by the automated routine. These data are automatically transferred to the "new_stdcal_data" and "new_He3cal_data" worksheets. Note that, although data are generated for the entire temperature range, you will limit the temperature range when you transfer the data.

   • **Save** the file and activate the "Stdpuck wire cond" worksheet.

   • Leave 1Point_Pass2_Data.xls open.

4. Puckxxx.cal file

   • Scroll down to the [Temp_Cond] section.

   • **Copy** the measured conductance (K_M) for the wire.

   • Leave Puckxxx.cal open.

5. 1Point_Pass2_Data.xls "Stdpuck wire cond" worksheet (Helium3 option: "He3Puck wire cond" worksheet)

   • **Paste** K_M into cell B2.

   • **Save** the file.

   • Activate the "new_stdcal_data" worksheet and **Save As** new_stdcal_data, selecting "Comma Delimited" as the File Type. (For the Helium3 Option, activate the "new_He3cal_data" worksheet and **Save As** "new_He3cal_data", selecting "Comma Delimited" as the File Type.)

   Note: The "Comma Delimited" File Type allows you to open the worksheet in

Notepad with the proper format for the Puckxxx.cal file; the file extension will be .csv.

- When you have completed these steps, close Excel and 1Point_Pass2_Data.xls but leave Notepad and Puckxxx.cal open.

Transferring the Data

Using the Notepad program, transfer the new heater-resistance data that was generated in Pass 2 (now in new_stdcal_data.csv or new_He3cal_data.csv) to the Puckxxx.cal file:

1. Puckxxx.cal file (If closed, open with Notepad)

    - Scroll down to the heater-resistance section, [Temp_HtrRes], and delete the original measured resistance at 305 K.

    - Leave the Puckxxx.cal file open.

2. new_stdcal_data.csv (or new_He3cal_data.csv)

    - Using the Notepad program, open the new_stdcal_data.csv file (or new_He3cal_data.csv).

    - In the heater-resistance section of new_stdcal_data.csv, [Temp_HtrRes], select your desired temperature range (the one you set up in Step 3 of Pass 1).

    - **Copy** the temperature dependence of the heater-resistance data.

    - Leave the new_stdcal_data.csv file open.

3. Puckxxx.cal file

    - **Paste** the temperature-dependence data into the [Temp_HtrRes] section of the Puckxxx.cal file.

    - Change the **Count** in the [Temp_HtrRes] section to match the new data.

    - **Save** the new Puckxxx.cal file.

4. Follow the same procedure to **Copy** and **Paste** the temperature dependence of the wire-conductance data from the [Temp_Cond] section of new_stdcal_data.csv (or new_He3cal_data.csv) to the [Temp_Cond] section of the Puckxxx.cal file.

5. **Save** and close the new Puckxxx.cal file.

6. Close new_stdcal_data.csv

7. When you next perform a heat capacity measurement, reference this new Puckxxx.cal file when you are asked to select a calibration file.

Figures 7–9 illustrate portions of the Puckxxx.cal file, including the calibrated data for the puck-thermometer resistance, puck-wire thermal conductance, and platform-heater resistance from 2.0 K to 20 K.

```
[Temp_PuckRes]

XFuncCode=2
YFuncCode=2
XName=Temp
YName=PuckRes
Count=23
2.000015,7332.1103
2.1982354,6214.1381
2.4014195,5360.5405
.
.
.
16.501813,649.78769
18.002595,604.92464
19.99969,555.12329
```

Figure 7. Example of calibrated data from 2 K to 20 K for the puck-thermometer resistance

```
[Temp_HtrRes]

XFuncCode=1
YFuncCode=0
XName=Temp
YName=HtrRes
Count=12
2.2375243,1807.3245
2.7696164,1760.884
3.490844,1714.9491
.
.
.
13.56279,1506.654
16.668117,1481.8567
20.201553,1459.5544
```

Figure 8. Example of calibrated data from 2 K to 20 K for the platform-heater resistance

```
[Temp_Cond]

XFuncCode=2
YFuncCode=2
XName=Temp
YName=Conductance
Count=12
2.2375243,2.3302284E-6
2.7696164,3.095517E-6
3.490844,4.2008579E-6
.
.
.
13.56279,2.4158585E-5
16.668117,2.9893548E-5
20.201553,3.5569236E-5
```

Figure 9. Example of calibrated data from 2 K to 20 K for the puck-wire thermal conductance