D403026

Advanced methods of solids characterization

K1: Puck calibration

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Development of new laboratory tasks for the DSP course D403026 "Advanced methods for solids characterization" and their translation into the English language

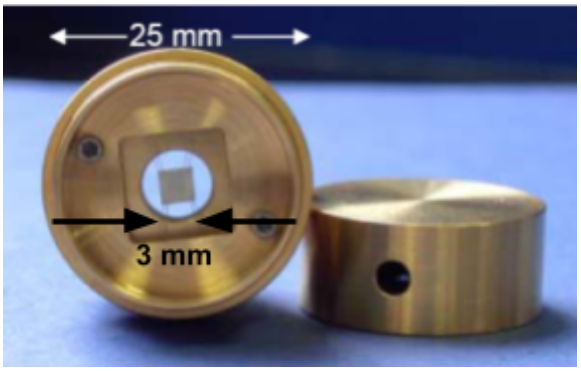
Content

Time frame and preparation for work

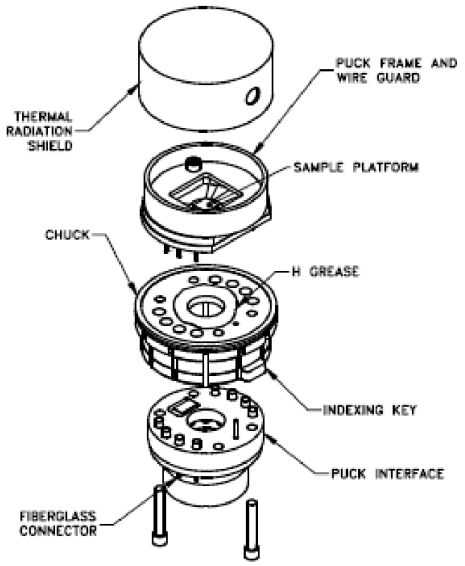
Prior to this task, it is necessary to complete the work “Determination of heat capacity by PPMS” or to at least get acquainted with the instructions for this work (the terms and activities explained in detail in this manual are explained very briefly here, e.g., control of MultiVu control program, etc.).

Before starting the work, it is necessary to become familiar with the WHOLE of this manual. When working, please wear gloves whenever you touch parts that are exposed to low temperatures.

Two “pucks” for measuring heat capacities were delivered to the PPMS.



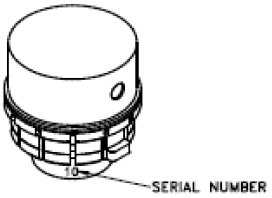
Photos of puck and its lids (left)



Exploded Puck Diagram (right; taken from Heat Capacity Option user’s manual)

Calibration consists of two steps, the first of which takes approximately 12 hours and the second approximately 8 hours. Operation of the device takes 2 hours for each step, and 1 hour for data evaluation.

1 Preparation for puck calibration



Before starting your own calibration, you need to:

a) find out the serial number of the calibrated puck, which is located on the bottom of the puck in green. In our case, these are the numbers 1676, 1677 and 2130.

b) remove the sample from the puck if there is one. Attaching and removing the sample from the platform can only be done after placing the puck in the so-called "mounting station", otherwise the puck will get damaged (repair costs nearly 1000 Euro). The use of the "mounting station" is described in Section 3.1 of the measurement instructions C_p[1].

c) remove the charcoal holder from the baffle assembly. This is done by unscrewing the silver thread from the gold cylinder located at the end of the plastic “rod”. An illustration of the heat shield system is shown in Figure A as Figure 4.6.

d) connect the cable marked “HEAT CAP” to the back of the PPMS and initiate the option “Heat Capacity” in MultiVu (if the cable is not connected and the option “Heat Capacity” has not been initiated). This activity is described in Chapter 3 of the manual[A] and is carried out by the supervisor.

e) set the temperature inside the well to 300.00 K and the magnetic field to 0.0 Oe. This is done by double-clicking with the left mouse button on the panel at the bottom of the MultiVu with information on the temperature and the magnetic field. This opens a dialog box where the temperature or magnetic field that we want to set can be entered. The panel along with the dialog boxes is shown in Fig. 1.1.

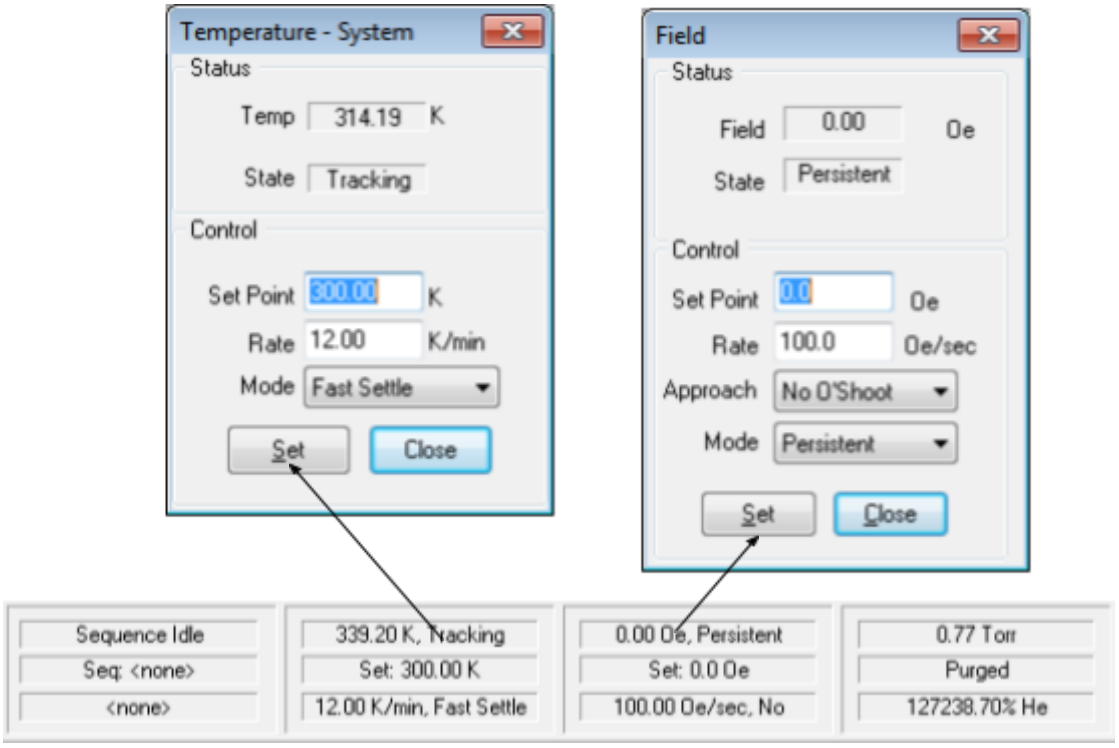


Figure 1.1: Temperature and magnetic field settings

[1] instructions "Determination of heat capacity by PPMS"

2 First part of calibration (Pass 1 of puck calibration)

In the first part of the puck calibration, called "Pass 1", the platform thermometer is calibrated. In this step, a .cal file is created, which contains information about the calibration of the puck and also stores data of the addenda measurements later on. The first part of the calibration is carried out with a small amount of helium in the measuring well to facilitate the thermal equilibrium between the individual thermometers in the system (the charcoal holder is initially removed from the baffle assembly for this reason).

All steps of the calibration are performed using a wizard, which is started by clicking on the “Prepare New Puck Calibration…” button in the HC option basic window - see Figure 2.1.

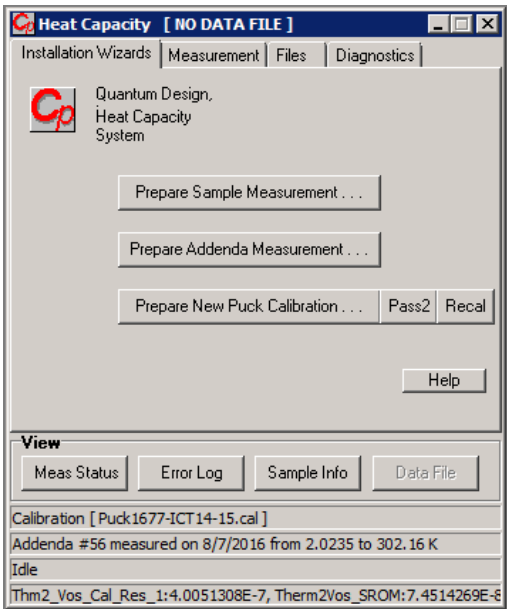


Figure 2.1: Start the puck calibration wizard by clicking the "Prepare New Puck Calibration..." button

The dialog box shown in Figure 2.2 will appear. At its top, labeled "Chamber Status", there is information about the temperature ("Stable" means 300 K is stable) and the well status ("Purged" means that the well was filled with helium, which was subsequently drained to clean it).

In the “Instructions” field, we are prompted to press the “Open Chamber” button to set the temperature to 300 K (if it is not already set). Then the measuring well is injected with helium to atmospheric pressure and can be opened. At the same time, helium is continually admitted for a maximum of five minutes to prevent air from entering the measuring well.

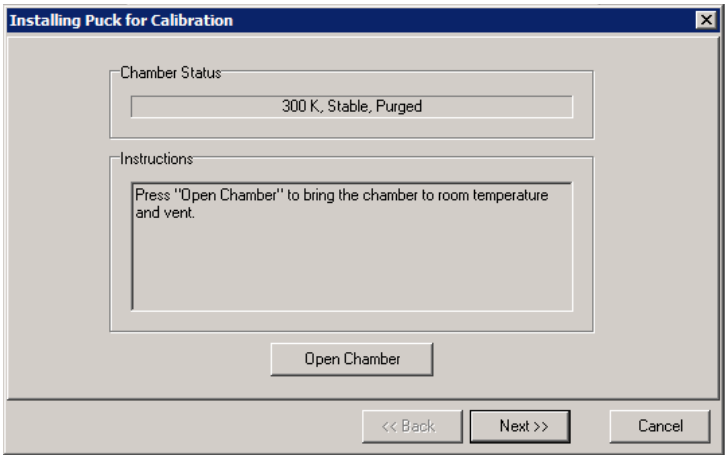


Figure 2.2: Pressing the “Open Chamber” button sets the temperature to 300 K (if it is not already) and the measuring well is sunk to atmospheric pressure, allowing it to be opened.

In the “Instructions” field, new information appears that lets the user know that helium is allowed into the measuring well (see Figure 2.3). We have to make sure that the charcoal holder has been removed from the baffle assembly. Once this has been taken care of, insert the puck (using the insertion mechanism, see Chapter 4.2 in the "Determining heat capacity using PPMS" manual) and the heat shield system in the measuring well and close it with the clamp. Then press the "Purge" button. During this step, the so-called “Cold Head” may stop. It will start again after a moment, but it is advisable to wait approximately 15 minutes for it to return to its original mode.

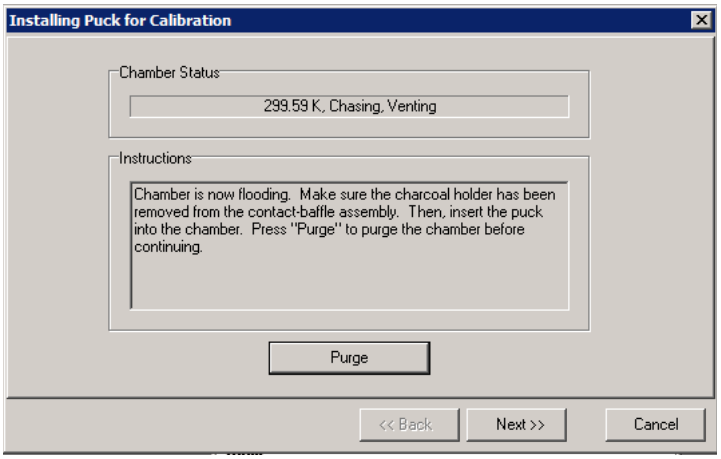


Figure 2.3: Instructions for inserting the puck into the measuring well

After pressing the “Purge” button, the “Instructions” field will display the command to select the “Next >>” button (see Figure 2.4).

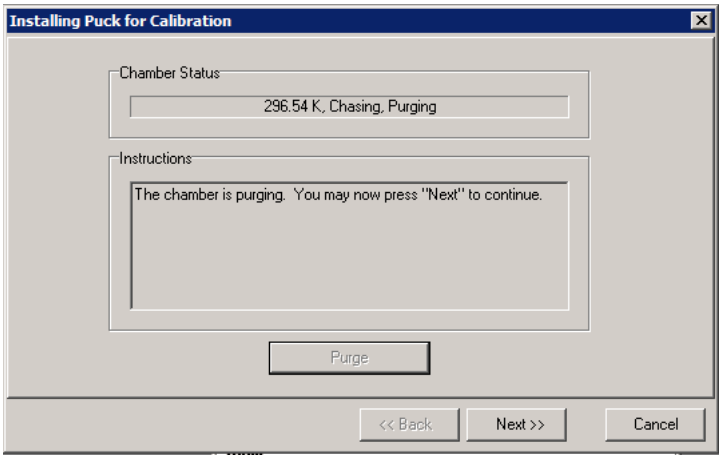


Figure 2.4: After pressing the “Purge” button, the measuring well is flushed with helium and it is possible to continue the calibration wizard by pressing the “Next >>” button

In the next dialog box (see Figure 2.5), enter the serial number of the puck located on the bottom of the puck, as well as the box in which it is stored (we have three pucks with serial numbers 1676, 1677 and 2130) and its type (Standard). After entering the required data, press the “Next >>” button.

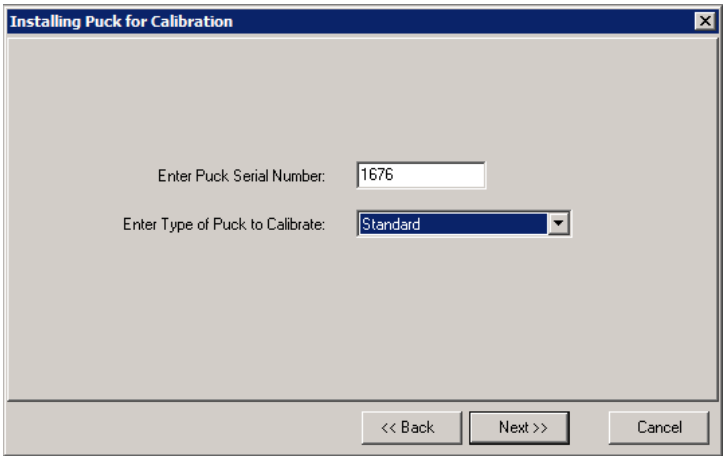


Figure 2.5: Instruction to enter puck serial number and type

A dialog box appears informing you that the puck is being tested (see Figure 2.6). If the test lasts for a few seconds, it is indicated by “Measuring… Please wait.” At the bottom of the window, if successful, the thermometer and heater resistance measured values will appear within the interval shown in the “Expected” field and the bottom of the window reads "Success!". If this does not happen, the puck is either incorrectly seated in the measuring well or is damaged.

If the puck testing is successful, press the “Next >>” button again.

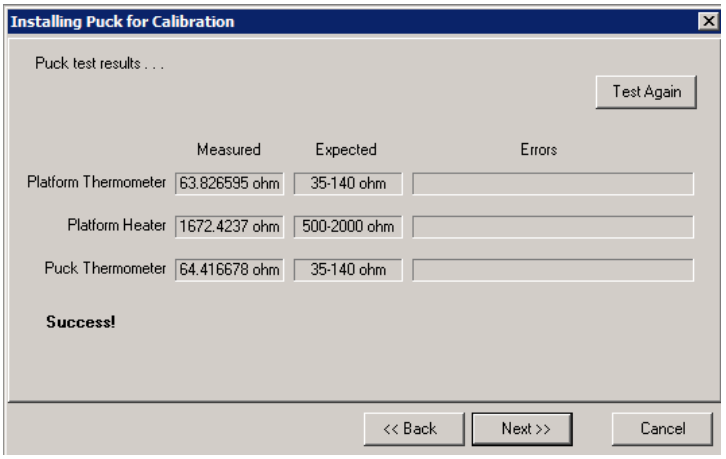


Figure 2.6: Information about puck testing in the measuring well

Now you have to enter the name and location of the file where the measurement data will be recorded. This is done by clicking on the “Open New File” button (see Figure 2.7). A dialog box the usually opens when saving files in Windows and in which you enter the path to the saved file (C:\QdPpms\Data\Heat Capacity) and its name (e.g., 1376-Puck1676-Pass1.dat, whereby the first four digits indicate the number of the PPMS experiment and the four digits for the word "Puck" indicates the serial number of the puck). After saving the file, the window shown in Figure 2.8 will appear. Click on OK to confirm. Then click on the “Finish” button in the dialog box in Figure 2.7.

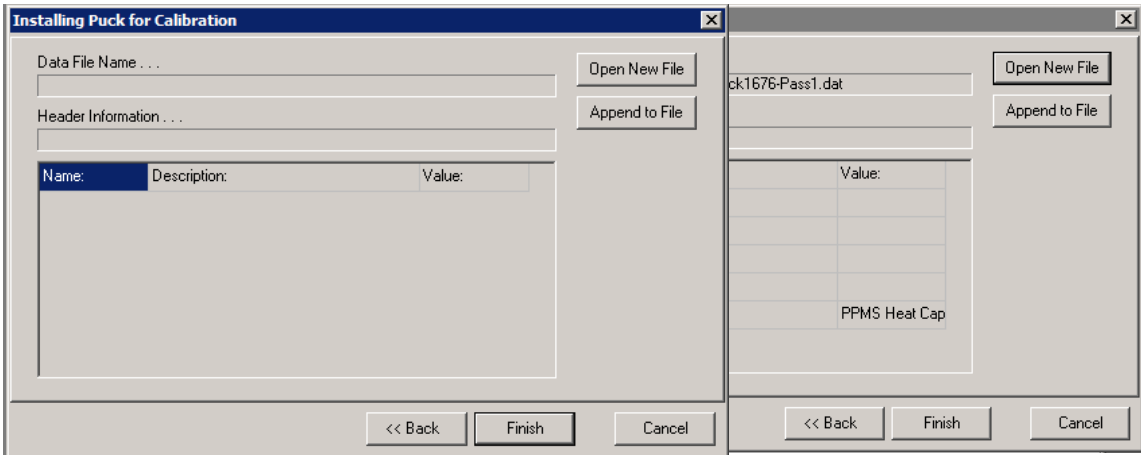


Figure 2.7: The window for entering the file information where the measurement data should be stored

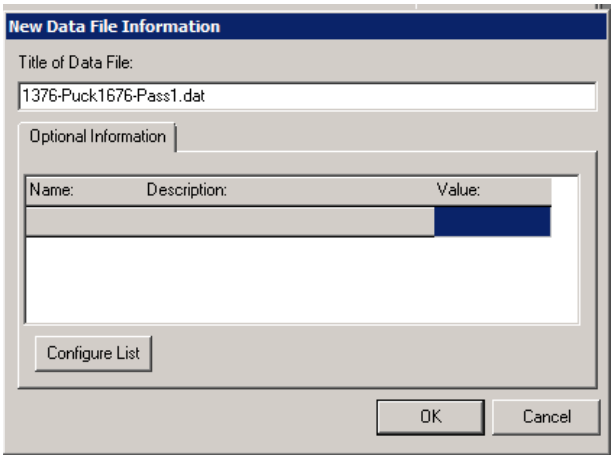


Figure 2.8: A dialog box that appears after entering the location and name of the file in which the measured data will be stored. We just approve it by clicking the "OK" button.

At the end of the first puck calibration step, the window shown in Figure 2.9 will appear, containing information about the serial number and type of the puck to remind you to remove the charcoal holder from the baffle assembly.

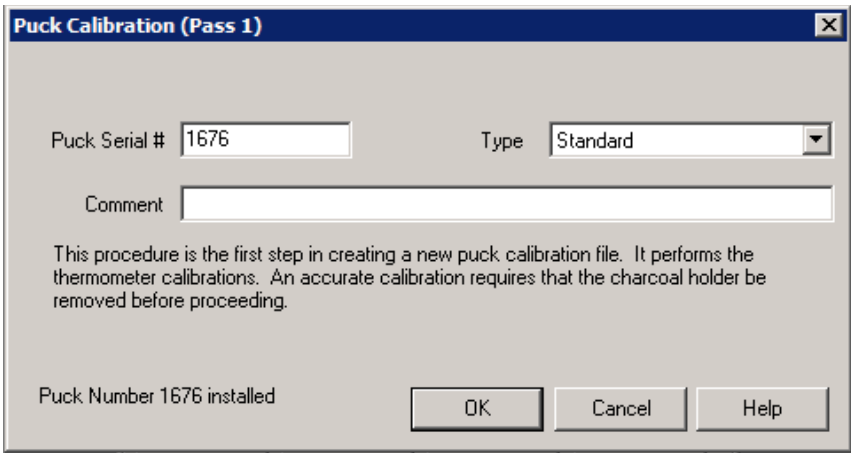


Figure 2.9: Summary of serial number and puck type information

After clicking on the “OK” button in the window in Figure 2.9, the first part of the calibration (Pass 1) will be started and the window in Figure 2.10 (“Measurement Status Viewer”) opens, which appears even in the case of measurement of thermal capacity but, at this time, remains blank in the item of “Total Heat Cap”, “Sample Heat Cap”, etc.

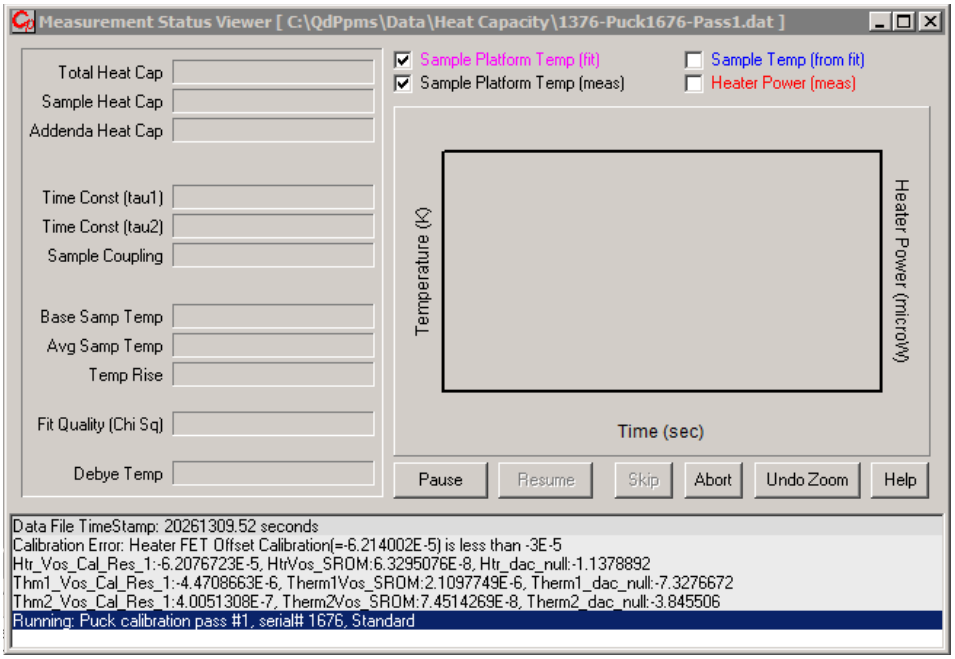


Figure 2.10: „Measurement Status Viewer“ - window in which the progress of the measurement is displayed

The first part of the puck calibration will take approximately 12 hours.

After completing the first part of the puck calibration, the system automatically heats up to 300 K.

After reaching the temperature of 300 K, it is necessary to wait about 90 minutes before the device can be opened without the risk of condensation of air humidity on cold parts of the apparatus.

3 Second part of calibration (Pass 2 of puck calibration)

After the thermometers on the puck are calibrated in the first part, the charcoal holder must be screwed back to the baffle assembly. This ensures the same pressure conditions (i.e., vacuum) in the measuring well as when measuring the thermal capacity of the sample or addendum itself.

The reason for performing the second part of the puck calibration is to measure the approximate value of the thermal conductivity of the wires connecting the platform to the puck and the electrical resistance of the heater on the platform. Although these exact values are not strictly necessary to determine the thermal capacity of the platform, tables of these values are used in conventional thermal capacity measurements to facilitate convergence of the nonlinear regression algorithm and to ensure that the temperature rise at the thermal pulse reaches the desired values.

After completing the first part of the puck calibration, the system automatically heats up to 300 K and the status of the measuring well changes to “Venting” (i.e., helium is blown in to prevent air from entering when the well is opened). If, at that time, the user is not present at the instrument to screw the charcoal holder onto the baffle assembly, a dialog box will appear five minutes after switching to the "Venting" status (see Figure 3.1). The sump ceases to be injected by helium so that it is not wasted and the status of the sump changes to "Sealed" (the valves in the sump for the helium inlet and outlet are closed).

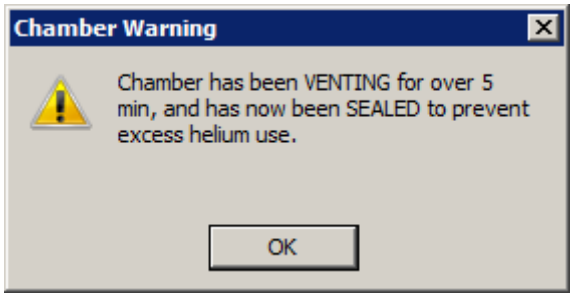


Figure 3.1: Dialog box informing about change of status of measuring well

However, if nothing happens, the admission of helium into the measuring well can be re-activated by clicking on the “Open Chamber” button in the dialog box shown in Figure 3.2.

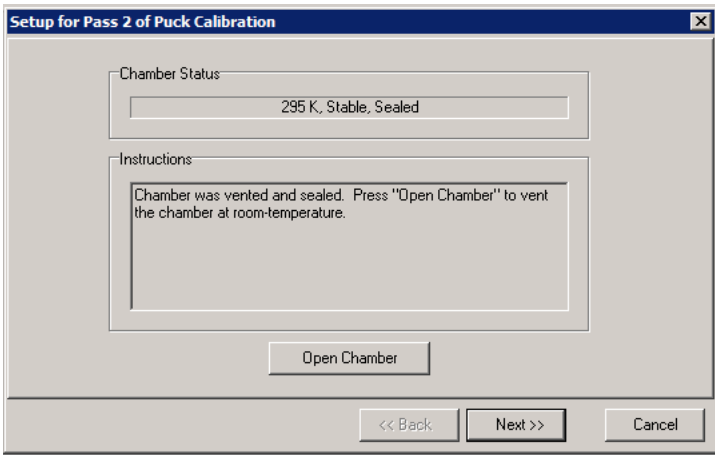


Figure 3.2: Clicking on the “Open Chamber” button will inflate the helium into the measuring well and open it.

After clicking the “Open Chamber” button, the status of the measuring well will change from “Sealed” to “Venting”. The "Instructions" field prompts you to reinstall the charcoal holder to the baffle assembly. First, remove the clip from the measuring well. Then, remove the heat shield system from which we screw the activated carbon holder, put it back in the well and secure it with the clip. Once done, click on the “Purge” button, which is shown in Figure 3.3. This will triple insert the measuring well with helium and drain it, thus removing any impurities from the well.

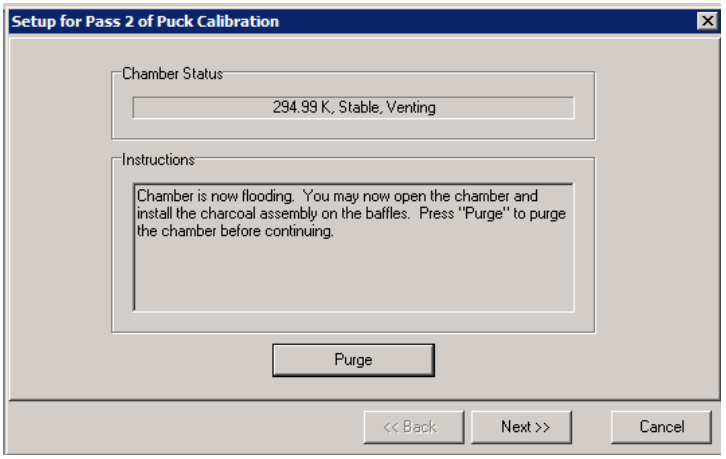


Figure 3.3: Invitation to screw charcoal holder onto baffle assembly

In the “Instructions” field, we are now prompted to click on the “Next >>” button (see Figure 3.4).

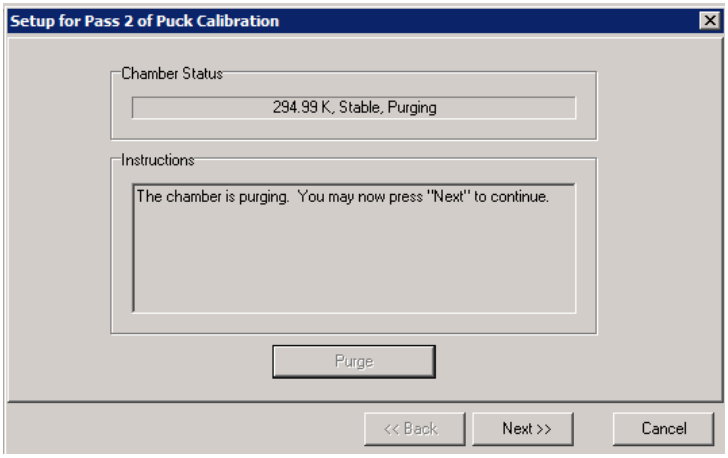


Figure 3.4: Dialog window asking to continue calibration („Next >>“ button)

Subsequently, the dialog box shown in Figure 3.5 (cf. Figure 2.6) appears, informing you that the puck is testing for a few seconds. If successful, “Success!” will appear at the bottom of the window (replacing “Measuring… Please wait.”).

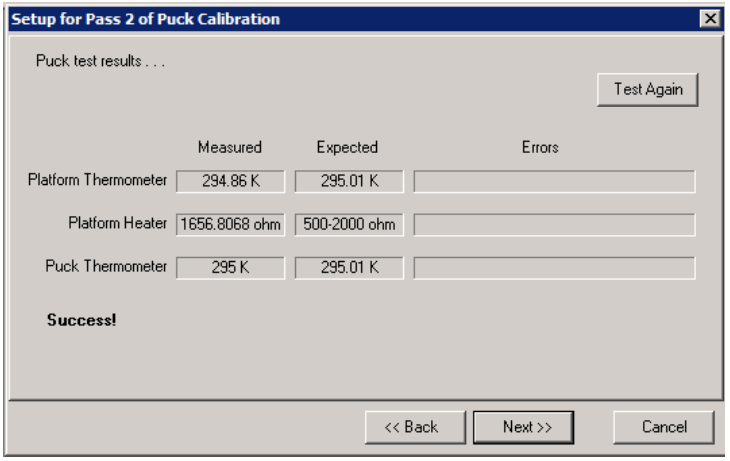


Figure 3.5: Dialog window informing about puck testing

If you continue by clicking on the “Next >>” button, a dialog box will appear, in which you can enter information about the location of the file where the measurement data will be saved and its name. This is done by clicking on the “Open New File” button (see Figure 3.6). Subsequently, a dialog box usually opens when saving files in Windows in which you enter the path to the saved file (C:\QdPpms\Data\Heat Capacity) and its name (e.g., 1376-Puck1676-Pass2.dat, with the first four digits indicating the number of the PPMS experiment and the four digits after the word "Puck" indicating its serial number). After saving the file, the same window as shown in Figure 2.8 will appear. Click on the “OK” button to confirm. Then click on the “Finish” button in the dialog box in Figure 3.6.

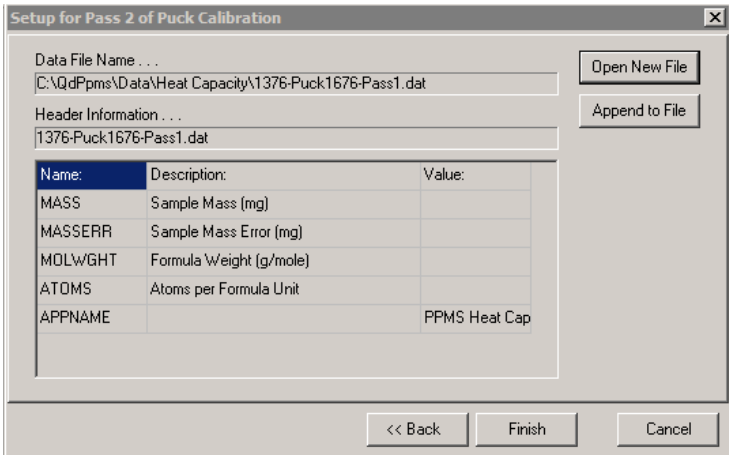


Figure 3.6: The window for entering the file information where the measurement data should be stored

The window for entering the measurement comment will appear (see Figure 3.7). This field can be left blank. Continue by clicking the "OK" button.

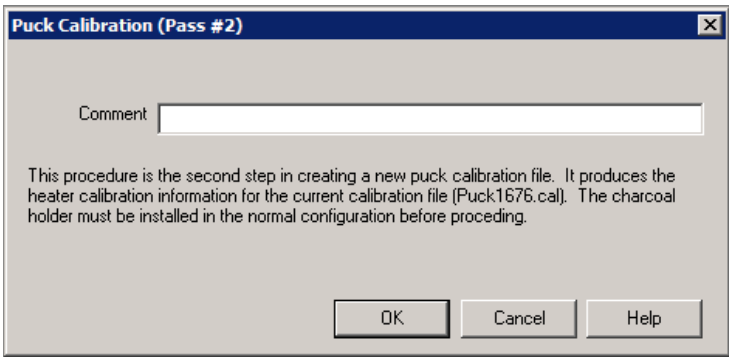


Figure 3.7: The window for entering the measurement comment. It is possible to leave it blank.

Finally, the second part of the puck calibration starts (see Figure 3.8).

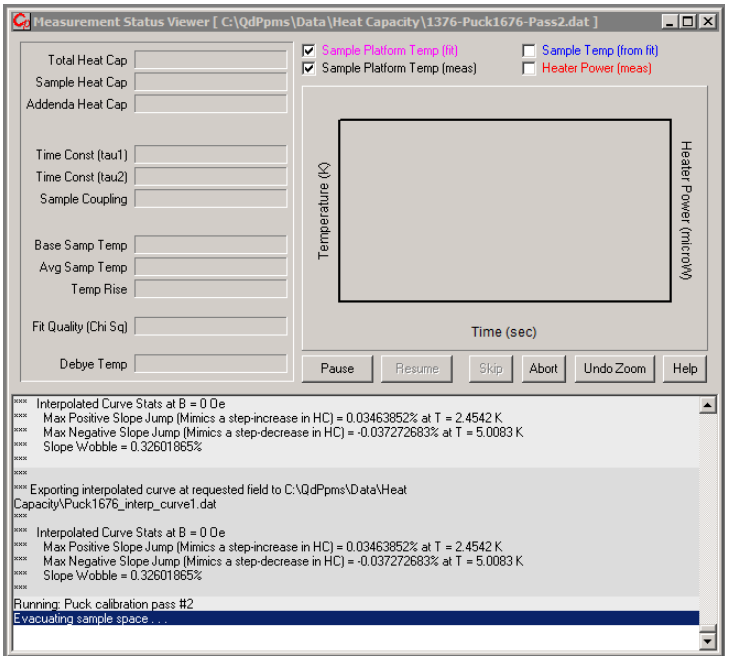


Figure 3.8: „Measurement Status Viewer“ - window in which the progress of the measurement is displayed

The second part of the puck calibration takes approximately 6 hours.

After completing the second part of the puck calibration, the system automatically heats up to 300 K.

After reaching a temperature of 300 K, it is necessary to wait about 90 minutes before the device can be opened without the risk of condensation of air humidity on cold parts of the apparatus.

4 Calibration check

Before starting to the puck calibration check, it is advisable to back up the newly created calibration file (preferably by copying it to another place on your computer).

After performing both steps to calibrate the puck, it is a good idea to check that the entire process has been successful. The first indication of a possible problem is the appearance of the error message shown in Figure 4.1.

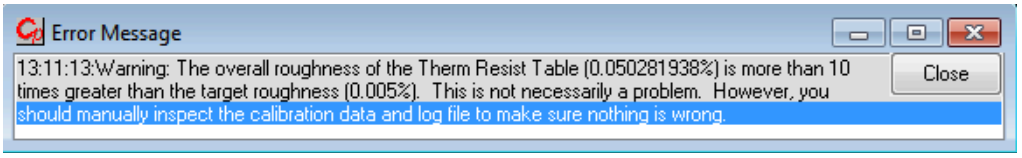
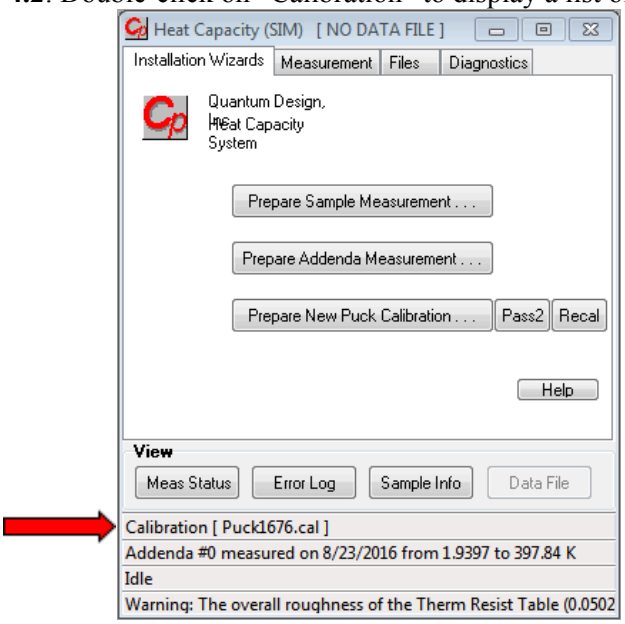


Figure 4.1: Error message indicating a possible problem during puck calibration

The user is prompted to review the calibration data to confirm or refute a potential problem. To display them, activate the window with the list of calibration files. This is done by double-clicking on the “Calibration” item at the bottom of the dialog box shown in Figure 4.2.

Figure 4.2: Double-click on “Calibration” to display a list of calibration files



A window with a list of calibration files is shown in Figure 4.3. The files must be located in the folder "QdPpms\Heat Capacity\TempCal\Standard" (they are stored in it automatically, knowing their location is important especially if you want to edit the calibration files on another computer in the so-called simulation mode (off-line evaluation).

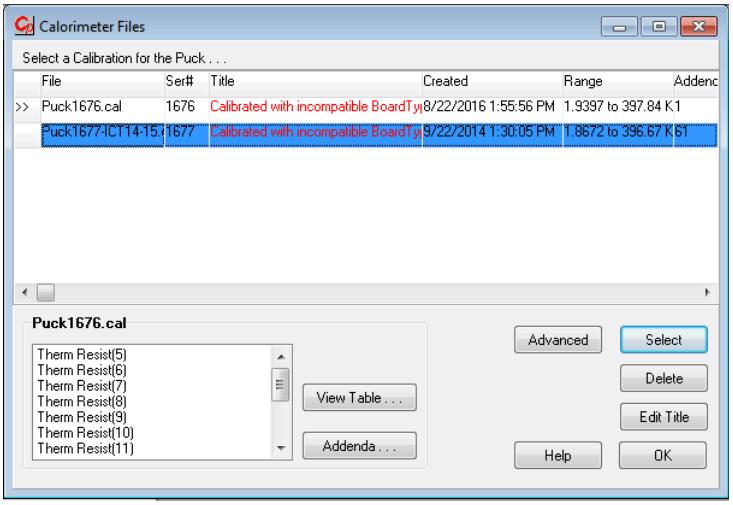


Figure 4.3: A window with a list of calibration files

The line with the active calibration file is marked with the symbol “>>” in the left part of the window. If you want to activate another calibration file, you have to click on the respective line with the mouse. They will turn blue and after clicking on the “Select” button a window will appear informing you that the calibration file has been activated (see Fig. 4.4).

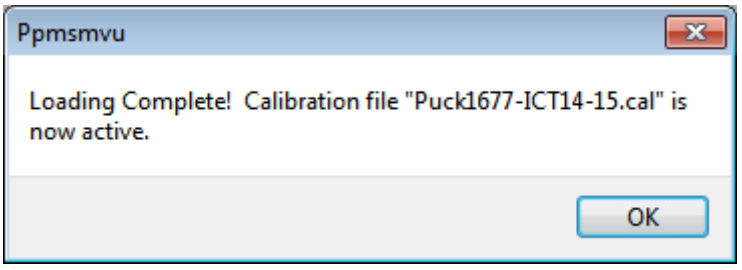


Figure 4.4: Information on activating the calibration file

In order to check the calibration of the puck, it is first necessary to activate the possibility of advanced operations with calibration files. To do this, click on the “Advanced” button and in the pop-up menu that appears, click on the only active item “Enable Advanced Table Operations” (see Figure 4.5).

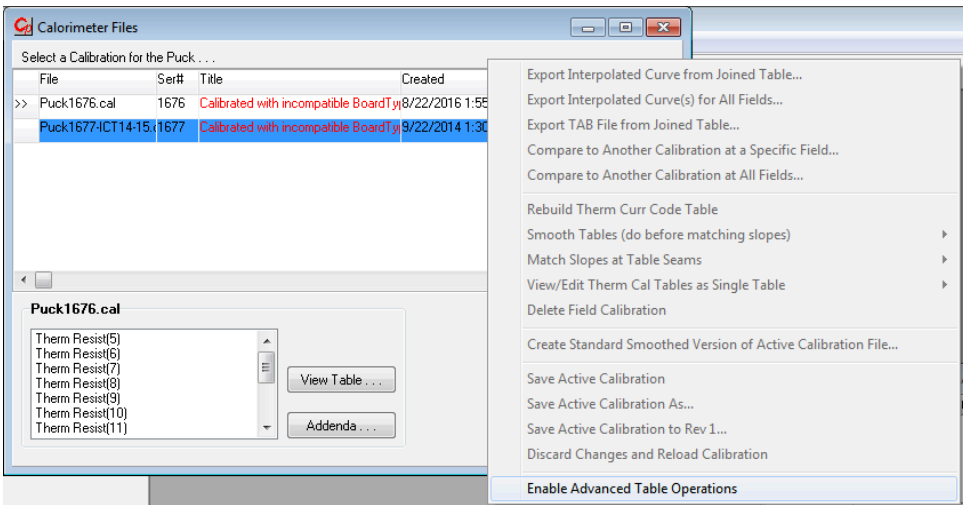


Figure 4.5: Enable advanced editing of calibration files

A window with confirmation of activation of advanced editing option will appear (see Figure 4.6), which can be confirmed by pressing the „OK“ button.

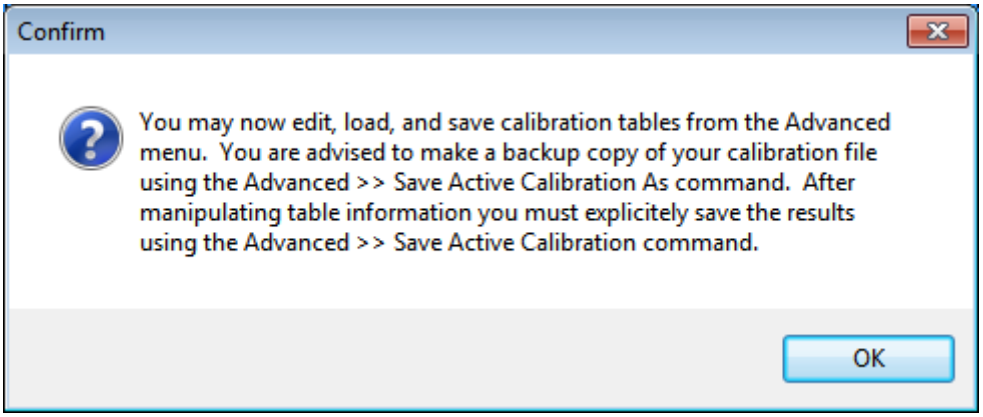


Figure 4.6: Confirmation of enabling advanced adjustment of calibration files

After enabling the advanced adjustment of calibration files, click on the “Advanced” button again and click on the “Export Interpolated Curve from Joined Table” item in the drop-down menu (see Figure 4.7).

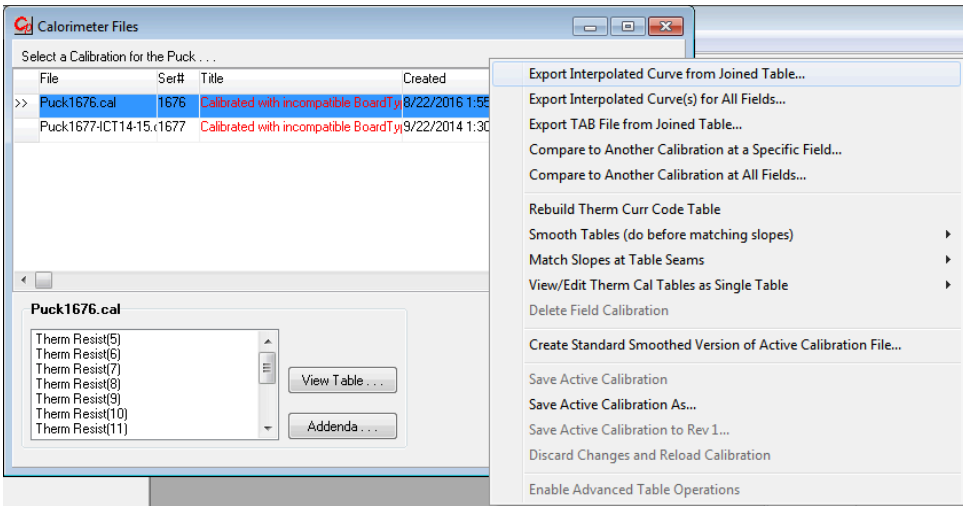


Figure 4.7: Selecting “Export Interpolated Curve from Joined Table…”

A window will appear (see Figure 4.8) where you enter the magnitude of the magnetic field at which the calibration was performed (in this case: 0 Oe) and click on the “OK” button.

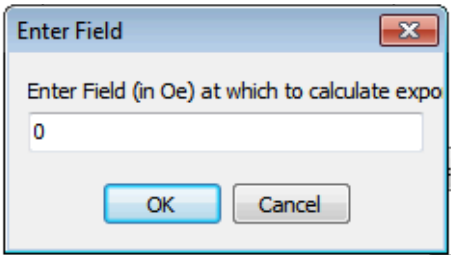


Figure 4.8: Enter the magnitude of the magnetic field at which the calibration was performed

This will open a dialog box that usually opens when you save files in Windows and specify the path to the saved file (C: \ QdPpms \ Data \ Heat Capacity). Keep the file name as written by the program in the file name line (eg "Puck1676_interp_curve.dat").

The saved file is then displayed in the graph in MultiVu - see Figure 4.9. According to the manual, no “bulges” or other phenomena should appear on the displayed curve, as this could indicate a poor calibration. The slope of the resistance versus temperature is directly proportional to the calculated heat capacity from the measured data. Figure 4.10 shows the same graph from puck calibration no. 1677 for comparison.

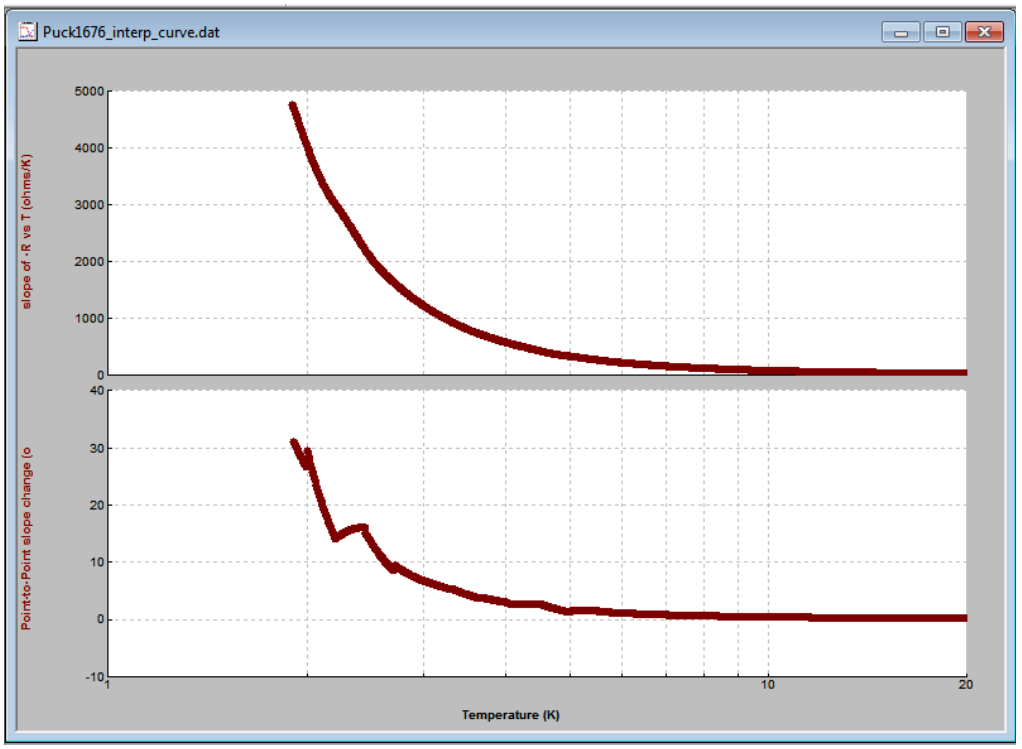
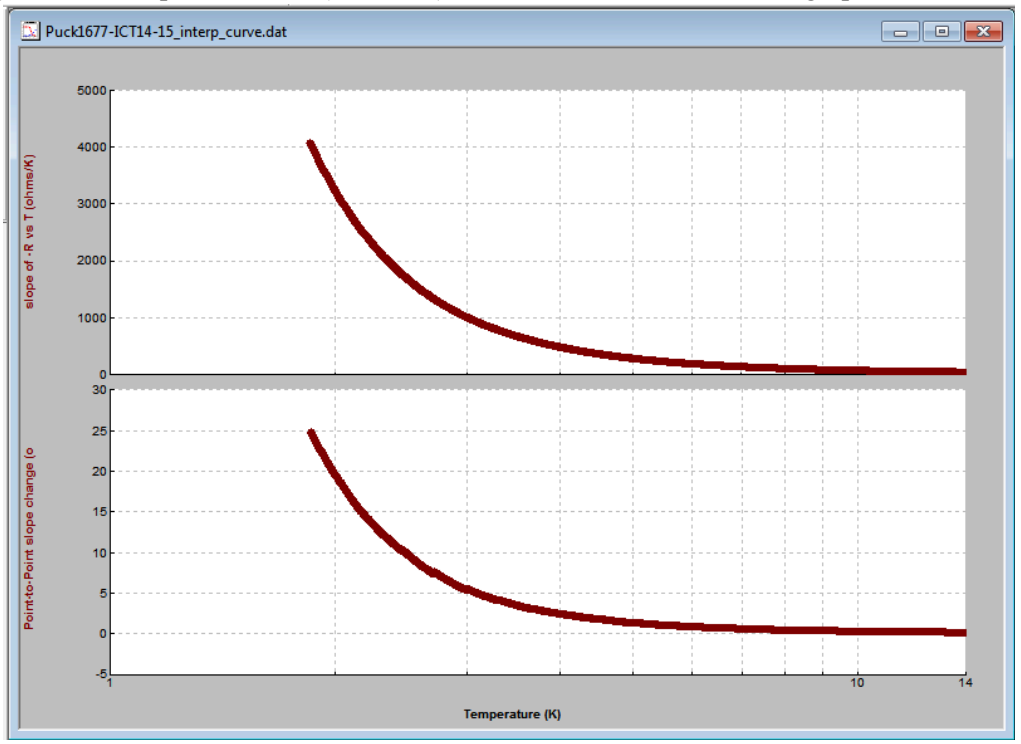


Figure 4.9: Slope of R vs. T (ohms / K), vertical axis name of bottom graph: Point-to-Point slope change (ohms / K) for Puck 1676.

Figure 4.10: Slope of R vs. T (ohms / K), vertical axis name of bottom graph: Point-to-Point



slope change (ohms / K) for Puck 1677.

This procedure can help us easily detect an unsuccessful calibration, but it is not sufficient to reliably check the accuracy of the calibration. For this purpose, it is best to measure the heat capacity of a calorimetric standard and to compare the results with literary values. For PPMS, copper proved to be the best calorimetric standard, for which Arblaster summarized and evaluated all available literature data and recommended an equation for the heat capacity with which we can compare our results. Above 50 K, they should not differ by more than 1 %, but below 50 K, deviations typically rise to 2-4% (see Figure 4.11).

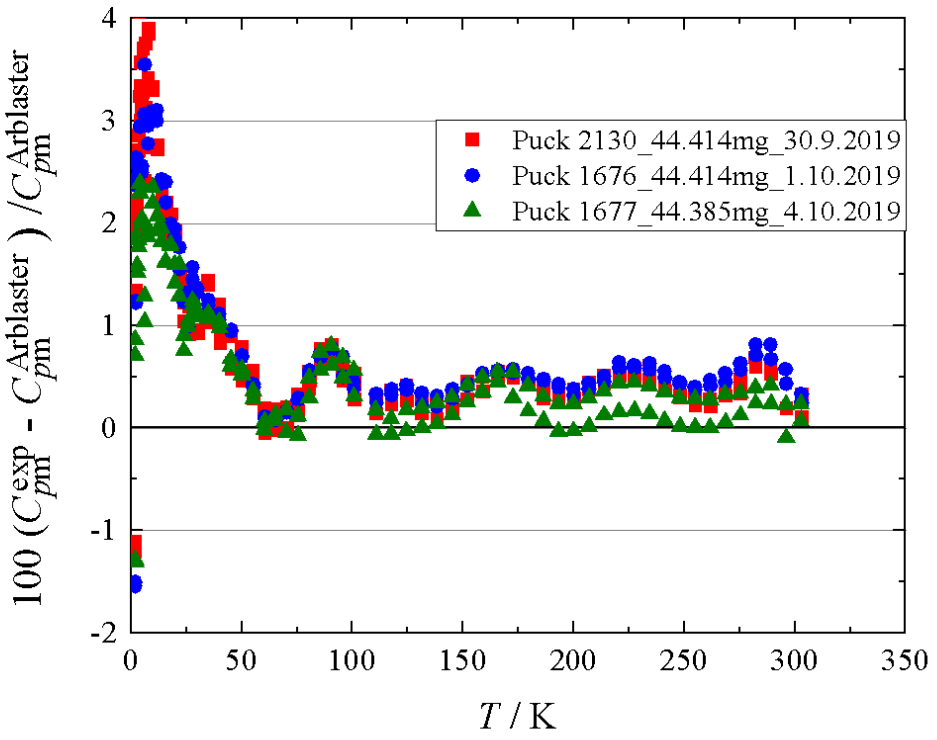


Figure 4.11: Deviations of copper heat capacity from the equation recommended by Arblaster for 2130, 1676 and 1677 pucks after calibration performed after service in September 2019.

5 Protocol

The report should include graphs from data in calibration tables. You can access this data by double-clicking on the “Calibration” item at the bottom of the dialog box shown in Figure 4.2. The “Calorimeter files” window opens with a list of calibration files. To the bottom left corner is a list of calibration tables contained in the selected calibration file. Select the desired table, press the “View Table…” button, select the “Edit” tab in the newly opened window, and copy the contents of the top window into the Excel table on this tab (procedure shown in Figure 5.1).

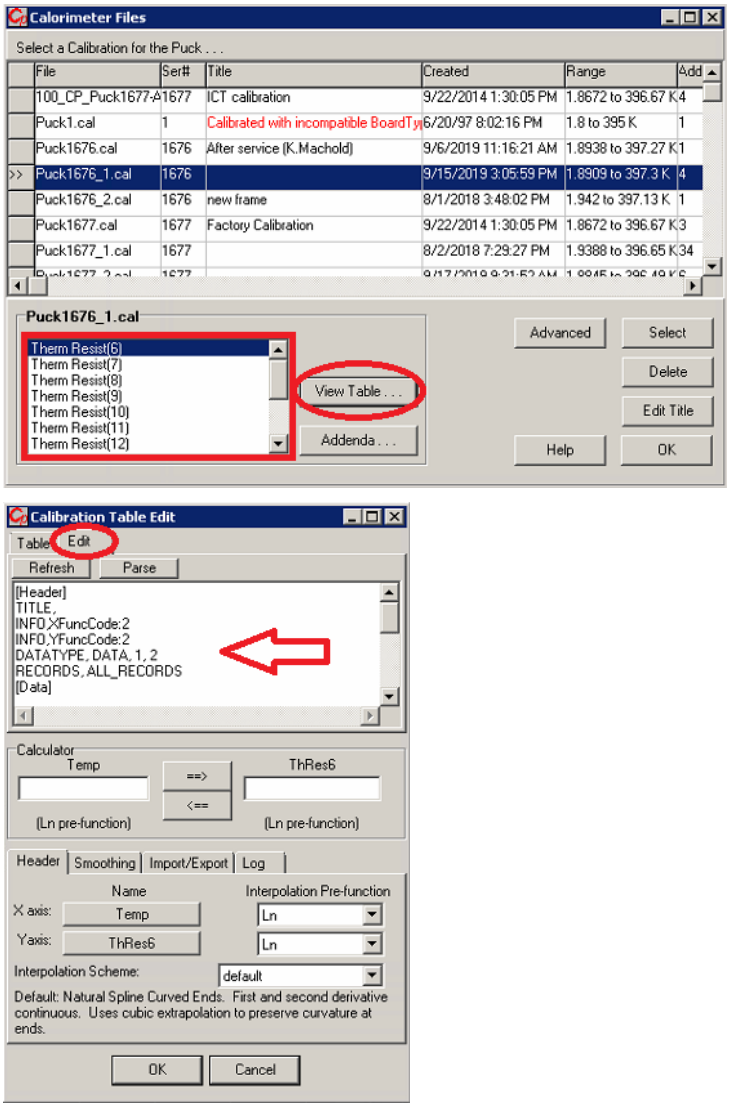


Figure 5.1: Procedure for extracting data from the calibration file

There are several “Therm Resist” tables in each calibration file, which are divided according to the excitation current that is used at given temperatures. These tables overlap and should be plotted in a common graph. Related to these tables is the "Therm Curr Code" table, which contains information on which "Therm Resist" table to use at a given temperature. We are also interested in the tables “Heater Resist” and “Thermal Conductance”.

In addition, the protocol should include the slope of R vs T and the point-to-point slope change graphs shown in Figures 4.9 and 4.10 on a logarithmic scale from 1 to 20. The procedure for obtaining them is described in Chapter 4.

If we measured a copper sample to check the accuracy of the calibration, we should also report a graph of copper heat capacity versus temperature and a graph of the thermal capacity deviation from the equation recommended by Arblaster (as in Figure 4.11).