Quantum Design

PPMS Application Note 1070-300

Method for Ensuring Monotonic Cooling of Samples to Temperatures near 2K in the PPMS

symptom: temporary loss of temperature control in some PPMS sample chambers at temperatures near 2 K after initially cooling from 300 K (see thin curve in figure), exposing the user's sample to a small thermal hysteresis.
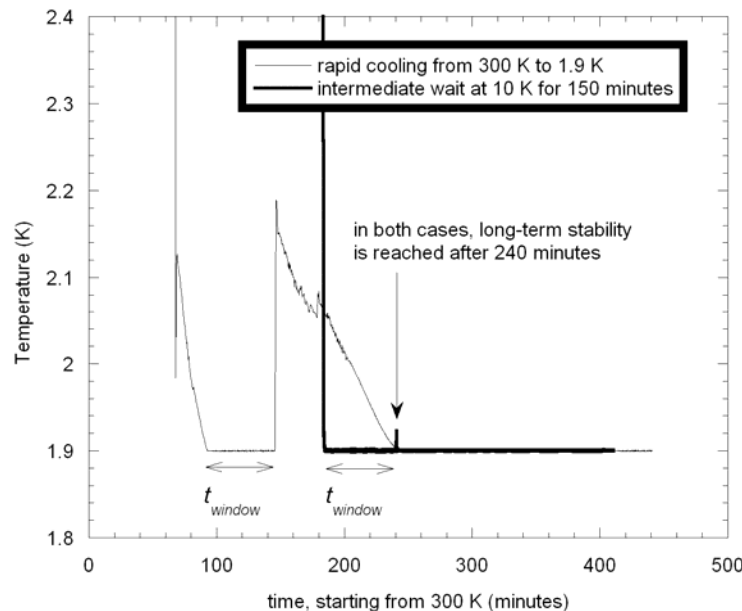

Figure 1: : time until stable temperature is achieved at 1.9 K in one particular PPMS system. Low temperature is approached either directly (thin curve) or using a wait at 10 K (thick curve). Variable t_{window} explained in text.

mitigation: ensure that the system spends at least a time t_{cool} below 50 K before reaching base temperature, where t_{cool} is determined as described below.

Introduction
The PPMS system provides fully automated temperature control over the whole temperature range from 1.9 K to 400 K and does this by using a dual impedance design. Smooth temperature control through the 4.2 K helium boiling point is achieved by closing off the high flow helium capillary ("main impedance") and relying on the secondary capillary ("continuous low temperature (CLT) impedance") to supply colder liquid helium to the cooling annulus around the sample chamber. A lower helium flow in

the CLT impedance, together with a low temperature setpoint near 2 K, mean that the cooling power of the system is much lower. A transient heat load is observed on all sample chambers after initially cooling from 300 K. This small heat load lasts for a few hours and for some systems it can be enough in this time period to overwhelm the cooling power when near 2 K, leading to a "bump" in the temperature and a slow recovery of temperature stability (see figure). Some samples can be strongly affected by thermal hysteresis such as occurs during this "bump". In these cases it is critical to find a protocol which preserves a monotonic temperature sweep. This note describes a method for assuring that.

Appearance of the Effect on a Given PPMS
There are a number of instrument-specific factors that can contribute to the appearance of the temperature "bump" in a PPMS. It is not a generic behavior, but given a known thermal history of the sample chamber and stable behavior of the helium impedances there is some predictability to the phenomenon. An example of this "bump" effect is shown in the figure above, where the thin curve corresponds to cooling from 300 K to 1.9 K at maximum rate. Temperature stability at 1.9 K is achieved around 90 minutes after initially cooling from 300 K, but is lost again near 140 minutes due to the above-mentioned transient heat load. The recovery to stable 1.9 K operation takes until a timestamp of 240 minutes. In contrast, the thick curve results if the sample chamber is held at an intermediate temperature of 10 K for 150 minutes. A small jump is seen but it is notable that long-term temperature stability is still achieved at 240 minutes. Some features to note in this data:

1) long-term temperature stability is only reached after 240 minutes, regardless of the details of the protocol.
2) the time between initial temperature stability and a "bump" is the same in both curves and is due to a small step-like decrease seen in the helium flow rate. Here, this value was about 50 minutes and this "stability window" value is referred to as t_{window}.
3) waiting for an intermediate amount of time at 10 K (60, 90, 120 minutes) results in "bumps" whose magnitude and duration decrease for longer wait times.

Note that this data is from a particular PPMS system and each instrument will have distinct temperature features as well as t_{cool} and t_{window} values. In fact, the temperature "bump" may be altogether absent on a particular PPMS.

Determining Cooling Time t_{cool}
In the data set from the figure, it took 30 minutes to get from 300 K to 50 K and overall required 240 minutes to get long-term stability at 1.9 K. Thus, we can state that the system needed to be below 50 K for 210 minutes to ensure stable temperature performance. So, for this PPMS $t_{cool} = 210$ minutes.
Procedure for quantifying t_{cool}:
1) insert a standard PPMS puck and baffle set in the sample chamber
2) purge the sample chamber
3) start a PPMS log: select 10 sec. logging interval and select all parameters in standard and diagnostic tabs
4) keep the chamber at 300 K for at least 4 hours

5) set 1.9 K at 10 K/minute in Fast Settle mode
6) wait 6 hours after initial stability is achieved at 1.9 K
7) stop the PPMS log
8) the time between reaching 50 K and reaching long-term stability at 1.9 K is t_{cool}

Mitigating the Effect
- When possible for the user's research, a general practice which saves time is to design the experiment so that measurements are made while cooling. Furthermore, if these measurements require the system to be in the range of 10 – 50 K for more than a time t_{cool} then the temperature "bump" will be avoided.
- If it is necessary to cool quickly and directly to low temperature, a measurement can be made at base temperature if it is shorter than the stability window t_{window}.

Ensuring Monotonic Cooling of the Sample Chamber
If it is critical to avoid any thermal hysteresis during cooling, then use this protocol to ensure a monotonic temperature trajectory:
1) Using No Overshoot mode to set the temperature, stabilize in the range T = 10 – 50 K for a time t_{cool} - t_{window} (=160 minutes here)
2) set T = 5 K in No Overshoot mode and wait there t_{window} + 10 minutes (=60 minutes here)
3) set the base temperature in No Overshoot mode

The intermediate temperature of 5 K is inserted so that any small flow step seen after being in low temperature mode for a time t_{window} (see thick curve in figure) will not cause temperature instability. This is because the cooling power of the system at 5 K is higher and is able to adjust to the flow change.

If there are further questions about the phenomenon, please contact apps@qdusa.com. For all other issues, please contact service@qdusa.com.