Quantum Design MPMS-3 Base System: Magnetic Field, Temperature, and Diagnostics

Magnetic field

- Field setting **resolution**: 0.33 Oe
- **Sweep rate** limits: 2 – 700 Oe/sec (note that for ultimate measurement sensitivity, SQUID data is collected at fixed field, not while sweeping field)
- **stability:** (QuickSwitch closed)
  - <0.1 Oe (short term)
  - +/- 1 Oe (drift from flux creep) in case of ramping linearly from a high field >1 kOe to low fields; see Fig. 4 in Application Note 1500-011 with this training document for ways to mitigate this
- **Uniformity:** at high fields (>1 T) it is very good: ± 0.01% over a 4cm vertical distance, centered on the SQUID detection coils; at low fields, due to field remanence issues it can vary by over 1 Oe over a DC Scan, depending on how the field was reached (this is important for measurements of, e.g., superconductors at low fields). See the "zero field Hall profiles MPMS3.xlsx" Excel worksheet and screen shot which are posted next to this document on the Wiki for specific profiles.
- "**Virgin" sample measurements not possible:** some materials require a measurement starting from zero applied field where the sample is in a virgin state (no history of magnetic field applied to it). Note that your sample will experience a vertical magnetic field, in both the positive and negative directions, of **+ and – 150 Oe** when inserting through the linear motor. If this is unacceptable, please consult Neil.

Accuracy above 1 tesla: better than 0.2%

Accuracy below 1 tesla: a systematic offset of up to 25 Oe exists, and is governed by magnet charging history. Note that the magnetic field value reported to the user is based only on the current flowing from the magnet power supply when the QuickSwitch was closed, and does not know about any other magnetic remanence effects near the sample. In our case, the superconducting coil itself remains magnetically polarized at low fields. The origin of this effect and various mitigation strategies are described in detail in the Application Note 1500-011 (see MPMS-3 Wiki page) and is also included in this training material.

Here are strategies for getting the best field accuracy in your measurements:

1. **If you are working at low fields only (H < 1000 Oe)**
   - Use the *Call Sequence* command at the beginning of your sequence and run `c\qdsquidvsm\sequence\magnet degauss from +3T.seq`
     "degaussing" means the following: set +3 tesla, wait for stability, then set zero field in Oscillate mode and wait for it to get there.
   - Insert your sample and do all your work at low fields: you will have accuracy of +/ 0.5 Oe if you stay below 1000 Oe at all times (orange box in lower plot of Figure 1). Note that

there is still a +2 Oe offset that remains in the magnet after oscillating down, so your loops will all be shifted to the right

2. If you need to use higher fields (H > 1000 Oe) and still care about +/- 25 Oe accuracy

- There is a simple correction applied in a script loaded at the MPMS-3 called `HighFieldSlope and HRemanence Correction.BAS` which will suffice for getting you within 1 Oe accuracy in most cases. You can find it in this folder at MPMS-3: *c\QdSquidVsm\Macros\Macros__QD_Library_Oct_2015\data postprocessing scripts* or find in MultiVu under *Utilities > Tools…* . More details about using the script are in the "interpreting the magnetic moment" document in this training series.
- if you select to correct magnet remanence in the script, it will a simple approach to shift ALL the reported fields DOWN (by the amount given in H_offset value) when coming DOWN in field, and UP when coming UP. It uses just the H_offset value from the maximum field visited since you degaussed the magnet. If working at low fields (<5000 Oe), the script assumes you ran the degauss procedure (described above) before measurements.
  - ⇒ The corrected field will be written to a new column of data called "[HFSS] H_corr (Oe)"
- This script will also subtract a slope from the data, typically for use in removing signal from a diamagnetic substrate.
- To understand the field correction visually: consult the upper plot in Figure 1 and use as follows: if you came down from +1 tesla (10000 Oe) then there will be a -25 Oe offset in the reported field. That is:
  - ⇒ H = H_reported + H_offset = H_reported - (25 Oe)
- Note that if coming up from -1 tesla, you will have a +24 Oe offset. The offset can be slightly different magnitude depending on the charging direction. This is very important if you are looking for a small exchange bias in your material and you need to use large fields for the loop.
- Another point about remanent fields: look at the bottom graph of Fig. 1 and see that the H_offset value goes from nearly zero at |H|<1000 Oe to over 20 Oe for fields |H|>5000 Oe. This is telling us something very important: magnetic flux penetrates into the magnet in the 1000 to 5000 Oe region and this causes issues for SQUIDs, leading to drifts in the SQUID voltage and causing artifacts like offsets in measurements.
  - ⇒ If measuring M(H) in this field region of 1000-5000 Oe, first ensure that these drifts are not important in your measurements by making several (like 5) *repetitions at each field* in the Moment v. Field dialog. If drifts are significant, you will see the reported moment value relaxing at a given field. You can filter the data later using the column *Measurement Number* (1,2…5 in the case of 5 repeats).
  - ⇒ Smaller field steps are another way of reducing the drift, if it continues to plague you.
  - ⇒ Magnitude of moment drift seen by doing repeated measurements in these conditions is small but important for some users: **typically ~2e-7 emu.**

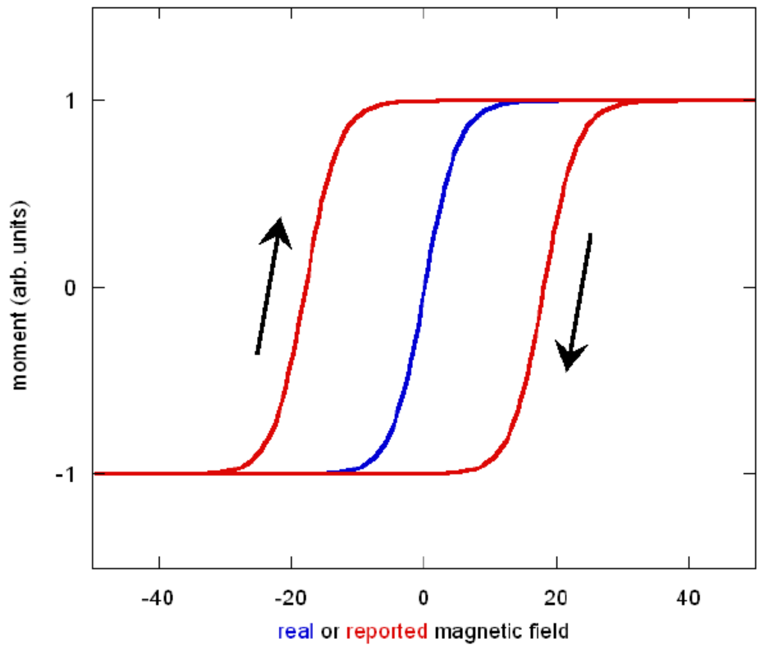
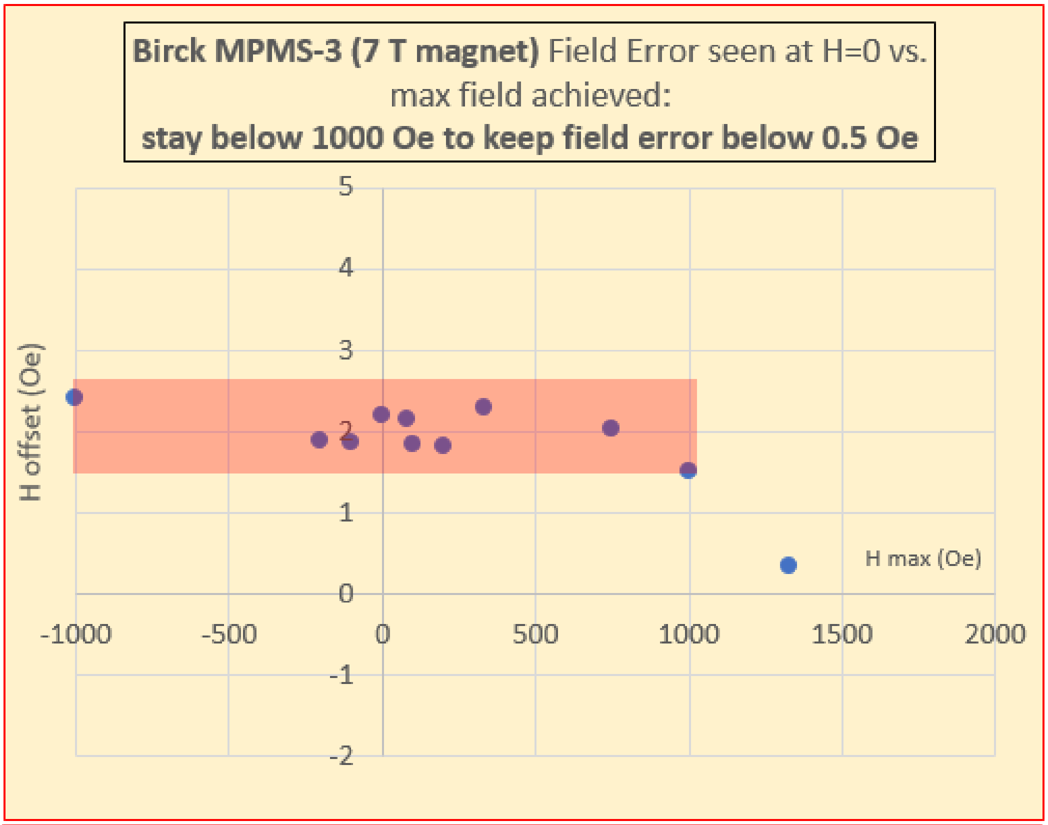


Figure 1: A magnetically reversible material (blue curve) can appear hysteretic (red curve) due to magnet remanence. ***Beware****! If apparent coercivity is on the order of 20 Oe, check that it is not "inverted" like this.*



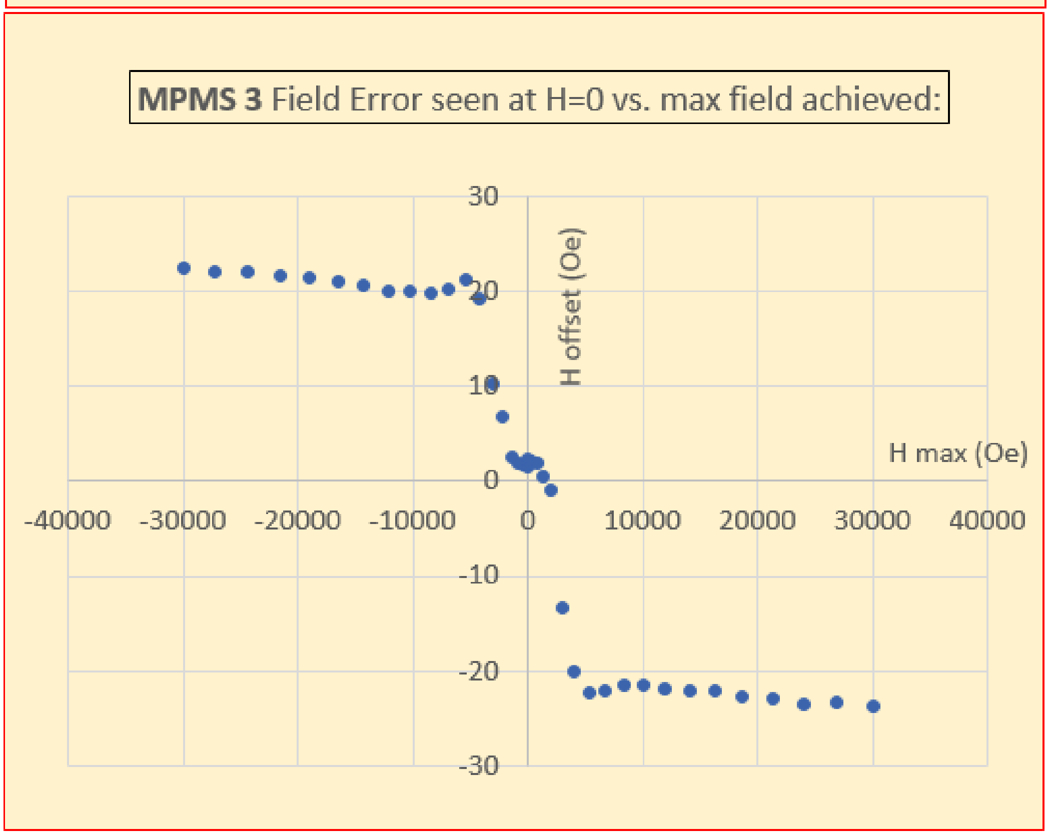


Figure 2: error in the reported field after OSCILLATING down from +3 tesla: low fields (TOP) and high fields (BOTTOM)

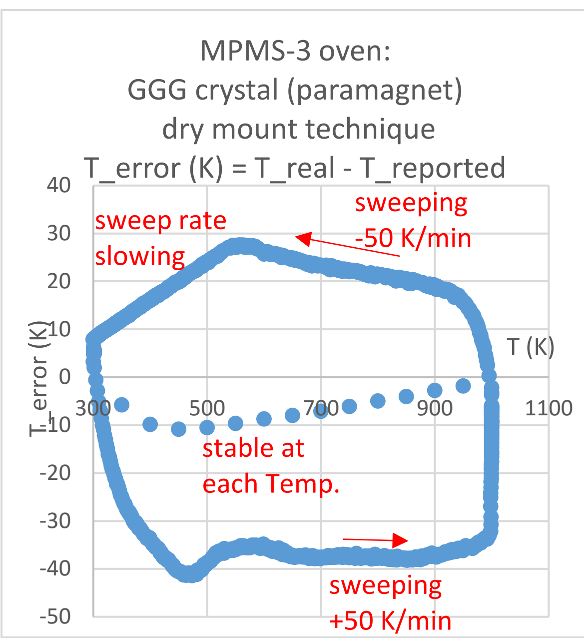
Temperature – oven mode (300 – 1000 K)

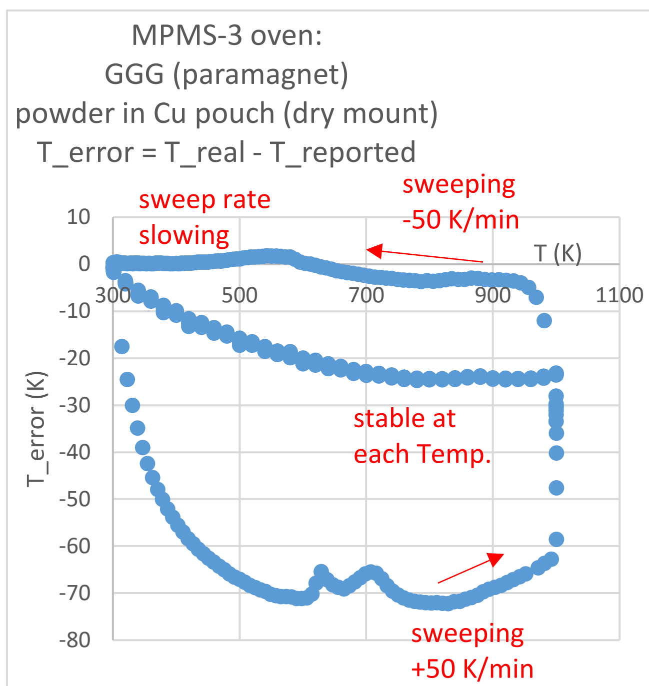
In the MPMS-3 oven, a thermocouple measures the temperature on the back side of the heated sample holder "heater stick" which is very close to the sample but due to large thermal gradients between the stick and the copper radiation shield we see significant 1-3% temperature errors depending on some factors. For best temperature accuracy, the following is recommended:

1) Flat, thin (<0.5mm thick) sample: couples best to stick, and fits under dry mount plate
2) Dry mount method using the ceramic plate and copper staples: compared to cement method, this is more reproducible, MUCH less wear on the stick, faster, easier to use
3) Clean copper foil radiation shield
4) Stabilize at each temperature (no sweeping), due to sample thermally lagging behind the heater stick thermocouple

The graphs below show temperature error for sweeping and stabilizing temp. throughout the oven temperature range. On the left is a solid crystal sample of GGG, while on the right is a powdered version of it which is packed and well-contained in a separate copper pouch. Not surprisingly, the crystal is better thermally coupled to the sample holder than the powder. Remember two factors here:

1) Thermal lag: the sample can lag up to ~60 sec behind the stick when sweeping temperature, and even more for powder-in-pouch sample.
2) Steady state Thermal gradient: even when stabilizing, the sample is a bit cooler than the stick (T_error < 0)





Temperature – standard mode (1.8 – 400 K)

The reported sample temperature comes from a thermometer reading that has been corrected for a number of factors, including magnetic field, thermometer location, thermal history, and thermal conduction from gas flow past the sensor. These corrections can only provide an approximation of the exact sample temperature. The most accurate sample temperature data is achieved at steady state. Dynamic corrections, and the associated errors, are smallest when the rate of temperature change is small. For more details on temperature control, read below...

The MPMS 3 sample chamber has a number of heaters and thermometers attached to the outside. Three Negative Temperature Coefficient (NTC) thermometers are used for temperature sensing. The primary sensor (sample thermometer) is located at the null point of the SQUID pickup coil. Two other sensors, gas and neck thermometers, are located at the bottom of the sample tube and between the sample thermometer and the top of the sample tube. Corresponding heaters are located near each thermometer. Because of the importance of the sample temperature accuracy, the temperature of the sample thermometer is calibrated in-situ in the system. Since strong magnetic fields can influence the accuracy of the thermometer at temperatures below 15 K, the sample thermometer is also calibrated under magnetic fields. The temperatures of the gas and neck thermometers are pre-calibrated prior to installation on the sample tube. The temperature control module reads out the gas thermometer, sample thermometer, and neck thermometer on the probe. The TCM also provides heater current to the gas heater, sample heater, and neck heater based on feedback temperature control using thermometer readings.

Temperature is regulated by PID (Proportional-Integral-Differential) control. The PID control is optimized depending on the temperature range of interest (Table 3-1). Between 320 K and 400 K, PIDs for the sample and gas thermometers are active. Going down in temperature from 320 K to near 10 K, the PIDs for the sample and gas thermometers are active. Going down from 10 K to 1.8 K and going up from 1.8 K to near 15 K, only the PIDs for the sample thermometer are active. Going up from 15 K to near 320 K, the PIDs for the sample and gas thermometers are used, while the PIDs for the neck thermometer is active between 100 K and 320 K.

Chamber Operations

IMPORTANT: In order to conserve helium gas and keep running costs of the machines down, please SEAL the chamber after VENTing in the Sample Install Wizard. That is, once you see the state being FLOODING please press the button to SEAL so that gas is neither being flooded into the chamber or being pumped out of it (Flooding state means the pressure is above 1 atm and He gas is exiting the chamber).

If the chamber is left in the flooding state, we can empty one UHP cylinder of He gas in 30 minutes, at a cost to us of $280!

Diagnostics

The measurement data file contains a wealth of information to help trace system parameters and possible issues with the measurement. Please read Appendix D in the MPMS-3 User Manual here.
In addition, a system log called `BRLog.dat` is always running the background (just like on the PPMS), and this is easily accessed in MultiVu under *Utilities > Tools > BRLog* .