



PPMS Application Note 1084-302

Calibration Procedure for the PPMS P500AC Susceptibility and DC Magnetization Option (ACMS)

The PPMS AC susceptibility and DC magnetization (ACMS) option is shipped with a Pd calibration sample for calibrating DC measurements. You can get Dy₂O₃ calibration samples from Quantum Design for calibrating AC measurements, also.

Calibration involves storing a response curve with known characteristics in a file for use by the ACDC-Mag software. Check DC calibration by comparing the Pd sample's measured magnetic moment to the theoretical moment at room temperature in a 20,000 Oe field. Check AC calibration by comparing the Dy₂O₃ sample's measured and theoretical AC magnetic moment amplitudes where the theoretical AC magnetic moment amplitude is obtained from two DC measurements. Quantum Design performs both calibration procedures before shipping the hardware, but you may want to re-calibrate if you suspect measurements are wrong.

Important: Recalibrating the ACMS involves deleting old calibration files and replacing them with new ones. Only experienced ACMS users should attempt recalibration.

These procedures require working ACMS hardware to be properly installed on the PPMS sample chamber and ACDC-Mag software to be properly installed on the PPMS computer. The two calibration samples also need to be properly mounted to sample holders in order to perform these procedures. **Calibration samples should be mounted using low susceptibility clear plastic straws.** DC calibration must be complete before performing an AC calibration. For those already familiar with the calibration procedure, a worksheet is provided at the end of this document for recording and calculating relevant values only. You may want to make copies of the worksheet. Four significant figures of precision are appropriate when performing the calculations below.

DC Calibration

1. Open ACDC-Mag in the PPMS directory.
2. From the Model 6000, set the temperature to 298 K. The ACMS user temperature (system bridge channel four) is the temperature of interest, so be sure that the **Temperature Control** display is set to **User Temperature** in the **ACDC-Mag Options** menu. User temperature is the default thermometer display when opening ACDC-Mag.
3. Set the field to 20,000 Oe from the Model 6000, using Oscillate mode.
4. Record the Pd sample's mass (provided on the sample box). $m = \underline{\hspace{2cm}}$ (g)

5. When the temperature reaches 298 K, select **Insert/Remove** from the ACDC-Mag **Sample** menu and insert the Pd sample.
6. Click the **finished** button once the sample is in the ACMS insert.
7. Select macro #21, **Center Sample**, from the **macro definition** box.
8. Set the **frequency** to 5 kHz and the **amplitude** to 10 Oe.
9. Once the PPMS temperature is stable at 298 K and the field is stable at 20,000 Oe, center the sample by clicking the button labeled **Run This Macro**. It is important to calibrate the system with a properly centered sample. If this step has not centered the sample well, do not proceed. Make sure the sample is properly centered by re-running the macro (remounting may be necessary in some cases) before continuing with the procedure. For more about centering see the *ACMS User's Manual*.
10. Select macro #25, **DC Measurement**, from the **macro definition** box.
11. Set the number of **Scans to Average** at 10.
12. Run the macro by clicking **Run This Macro**.
13. When the **Last result** box is updated, record the applied **DC Field** as H.
14. $H = \underline{\hspace{2cm}}$ (Oe).
15. Determine the Pd sample's theoretical moment using $M_{\text{theor}} = \chi_g mH$, where the Pd has mass susceptibility $\chi_g = 5.25 \times 10^{-6}$ emu/Oe-g, m is the sample's mass in grams and H is the applied field in Oe. $M_{\text{theor}} = \underline{\hspace{2cm}}$ (emu).
16. Select **Calibration** from the **Options** menu and choose **Save DC Moment...**
17. Save the theoretical DC moment calculated in step 14 above by entering the value (M_{theor}) in the pop up box and clicking **okay**.
18. Click **yes** when asked to save the waveform. The moment and waveform are stored in a file called DC-EMU.CAL and used for all subsequent DC measurements until a new value and waveform are saved. This file should not be deleted or tampered with.
19. Click **yes** when asked to use the new calibration immediately.
20. From the **Last result** display, record the reported moment from the DC measurement. $M_{\text{reported}} = \underline{\hspace{2cm}}$ (emu)
21. Calculate percent difference in moment: $\% \Delta M = \left| 1 - \frac{M_{\text{reported}}}{M_{\text{theor}}} \right| \times 100 = \underline{\hspace{2cm}} \%$
22. If $\% \Delta M < 0.25\%$, DC calibration is complete. Otherwise, perform steps 23-24 until $\% \Delta M < 0.25\%$. Up to four iterations may be required. If the 0.25% tolerance is not reached, contact Quantum Design.
23. Run the **DC measurement** macro (macro #25) again by clicking **Run this macro**.
24. Recalculate $\% \Delta M$ using the new M_{reported} value.

AC Calibration

1. Set the system temperature to 298 K and allow the system to stabilize.
2. With the PPMS temperature stable at 298 K, use the **Insert/Remove...** option of the **Sample** menu to load the Dy₂O₃ sample.
3. Once the sample is loaded, click the **Finished** button.
4. Set the field to 10,000 Oe and wait for the field to stabilize.
5. Set macro #21, **Center Sample**, for **frequency** = 5 kHz and **amplitude** = 10 Oe and DC Centering.
6. Center the sample by running the **Center Sample** macro (macro #21). It is important to calibrate the system with a properly centered sample. If this step has not centered the sample well, do not proceed. Make sure the sample is properly centered by re-running the macro (remounting may be necessary in extreme cases) before continuing with the procedure. For more about centering see the *ACMS User's Manual*.
7. Select macro #25 to perform a DC measurement.
8. Set the number of **Scans to Average** at 10.
9. Run the Macro by clicking the button labeled **Run This Macro**.
10. When the **Last result** box is updated, record the result of the moment measurement as M₁. M₁ = _____ (emu).
11. Record the field for the previous measurement as H₁. H₁ = _____ (Oe).
12. Set the field to -10,000 Oe and wait for the field to stabilize.
13. Perform another DC measurement by running macro #25 again.
14. When the **Last result** box is updated, record the result of the moment measurement as M₂. M₂ = _____ (emu).
15. Record the field for the second measurement as H₂. H₂ = _____ (Oe).
16. Set the field to zero Oe.
17. Calculate $\chi_{ac} = \Delta M / \Delta H = (M_1 - M_2) / (H_1 - H_2) =$ _____ (emu/Oe).
18. Calculate the AC moment's theoretical amplitude in a 10 Oe AC applied field using $M_{\text{amplitude theor}} = \chi_{ac} H_{\text{amplitude}} = \chi_{ac} \times 10 \text{ Oe} =$ _____ (emu).
19. Select macro #20 to perform an AC measurement.
20. Set **Amplitude** = 10 Oe, **frequency** = 1 kHz, **acquisition time** = 10 sec and **record harmonics** = fundamental.
21. Wait for the field to reach zero Oe. With the field stable at zero Oe, run macro #20.
22. From the **Options** menu, select **Calibration**, and then **Save AC Moment...**
23. In the box labeled **Known value**, enter the moment's theoretical amplitude calculated in step 18 above.

24. In the box labeled **This change authorized by**, type your name and the date, then click **Accept**.

25. Run macro #20 again to perform another AC measurement.

26. Record the moment amplitude from the latest scan as $M_{\text{amplitude meas}}$.

$M_{\text{amplitude meas}} = \underline{\hspace{10em}} \text{ (emu)}.$

27. Calculate the percent difference in moment amplitudes using:

$$\% \Delta M_{\text{amplitude}} = \left| 1 - \frac{M_{\text{amplitude meas}}}{M_{\text{amplitude theor}}} \right| \times 100 = \underline{\hspace{2em}} \%$$

28. If $\% \Delta M_{\text{amplitude}} < 0.25\%$, AC calibration is complete. Otherwise, perform steps 30 and 31 until $\% \Delta M_{\text{amplitude}} < 0.25\%$. Up to four iterations may be required. If the 0.25% tolerance is not reached, contact Quantum Design.

29. Run macro #20 to perform an AC measurement again.

30. Recalculate $\% \Delta M_{\text{amplitude}}$ using the new $M_{\text{amplitude meas}}$ value.

ACMS Calibration Worksheet

DC Calibration [Pd sample]:

$$\chi_g (\text{Pd}) = 5.25 \times 10^{-6} \text{ emu/Oe-g}$$

$$m = \underline{\hspace{2cm}} \text{ (g)}$$

$$H = \underline{\hspace{2cm}} \text{ (Oe)} \sim [20,000 \text{ Oe}]$$

$$M_{\text{theor}} = \chi_g mH = \underline{\hspace{2cm}} \text{ (emu)}$$

$$M_{\text{reported}} = \underline{\hspace{2cm}} \text{ (emu)}$$

$$\% \Delta M = \left| 1 - \frac{M_{\text{reported}}}{M_{\text{theor}}} \right| \times 100 = \underline{\hspace{2cm}} \%$$

$$\% \Delta M < 0.25\% ?$$

Yes: calibration complete.

No: repeat measurement, recalculate $\% \Delta M$.

AC Calibration [Dy_2O_3 sample]:

$$M_1 = \underline{\hspace{2cm}} \text{ (emu)}$$

$$H_1 = \underline{\hspace{2cm}} \text{ (Oe)} \sim [10,000 \text{ Oe}]$$

$$M_2 = \underline{\hspace{2cm}} \text{ (emu)}$$

$$H_2 = \underline{\hspace{2cm}} \text{ (Oe)} \sim [-10,000 \text{ Oe}]$$

$$\chi_{ac} = \frac{M_1 - M_2}{H_1 - H_2} = \underline{\hspace{2cm}} \text{ (emu/Oe)}$$

$$M_{\text{amplitude theor}} = \chi_{ac} \times 10 \text{ Oe} = \underline{\hspace{2cm}} \text{ (emu)}$$

$$M_{\text{amplitude meas}} = \underline{\hspace{2cm}} \text{ (emu)}$$

$$\% \Delta M_{\text{amplitude}} = \left| 1 - \frac{M_{\text{amplitude meas}}}{M_{\text{amplitude theor}}} \right| \times 100 = \underline{\hspace{2cm}} \%$$

$\% \Delta M_{\text{amplitude}} < 0.25\%$?

Yes: calibration complete.

No: repeat measurement,

recalculate $\% \Delta M_{\text{amplitude}}$.