Accuracy of Reported Sample Moment: Using the Sample Geometry Simulator

This Application Note discusses the accuracy of the sample moment reported by MPMS 3, common factors that introduce systematic errors to the reported moment, and techniques for improving the accuracy of the reported moment using the MPMS 3 Sample Geometry Simulator, which is available on the Pharos library.

Moment Accuracy

The accuracy of the reported moment has always been of great importance for MPMS users, especially when the moment values are used as inputs for further quantitative analysis, for example: a Curie-Weiss law fit to the susceptibility. Accuracy is also important when comparing the moment values obtained from different magnetometers, such as the MPMS XL, MPMS 3, and PPMS VSM.

The MPMS is a highly sensitive and precise SQUID-based magnetometer. However, as with any other experimental measurement apparatus, systematic errors can readily affect the reported moment values. Depending on how knowledgeable and diligent the operator is with the measurement process and sample mounting, the difference between the reported and the "true" moment can easily range from less than 1% to larger than 10%.

The measurement process of the MPMS has been thoroughly discussed in previous QD Application Notes (1014-214; 1014-822) and a recent book chapter **[1]**. In simple terms, the measurement involves moving a sample though the second order gradiometer, thus inducing a position- (DC-scan) or time- (SQUID-VSM) dependent voltage waveform, which is in turn proportional to the magnetic moment. A few key factors that affect the accuracy of the reported moment include:

- Sample mounting (Notes 1014-201, 1096-306)
- Background/inhomogeneity of the sample holder (1014-213, 1500-023, SQUIDLab **[2]**)
- Sample centering, both longitudinal and radial (1500-010)
- Sample size/shape (1500-015)
- Magnetic field accuracy (1500-011, 1500-021)
- Demagnetization effects (see, R. Goldfarb [1992])

Demagnetization effects are unique in that they involve a correction to the magnetic field axis while all others are corrections to the reported moment. It should be added that demagnetizing effects are primarily relevant in materials in regions of high susceptibility[1] (i.e. ferromagnets and superconductors).

[1] $\chi = dM/dH$

Centering accuracy and sample size/shape have the most significant effects on moment error **[3]**. Centering matters in both the longitudinal and radial directions (Figure 1). The longitudinal centering is performed automatically by the system. However, when the sample signal is small at room temperature, for example a thin magnetic thin film on a substrate, user judgment is required to correctly center the sample with the help of the sample mounting station. Radial centering on the other hand is less obvious and often overlooked. A quick way to check the radial centering is to rotate the sample rod and take measurements at a several (~6) angular positions (Figure 1). A large variation of the reported moment with angle indicates a large radial offset of the sample. The angular position with a *minimum* moment value is likely to be closest to the center of the sample chamber.

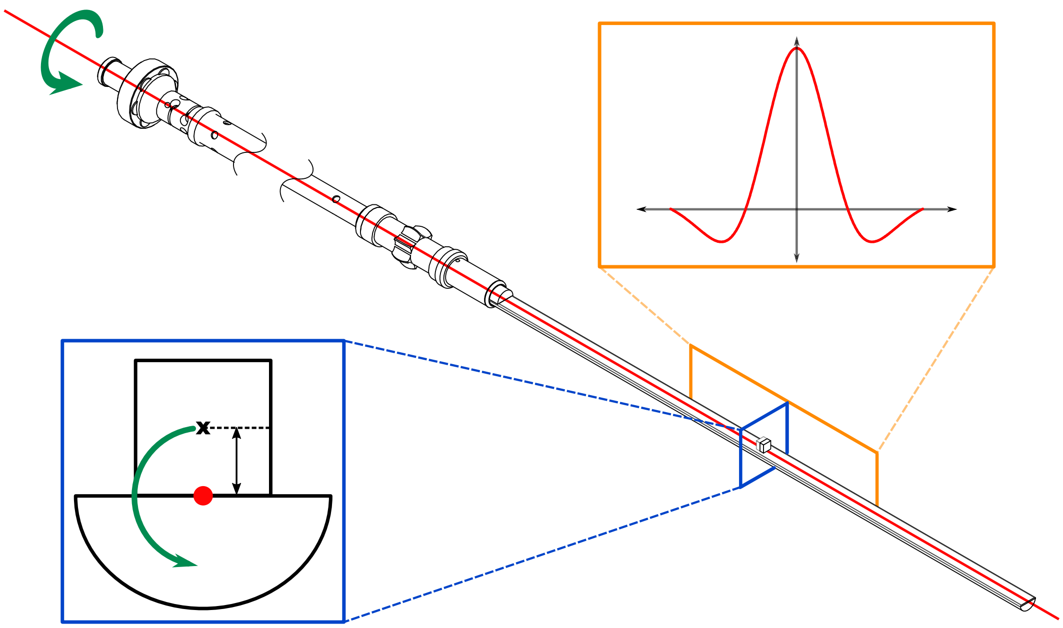

Figure 1: *Sample centering geometry: longitudinal (red) and radial (green).*

The Sample Geometry Simulator (SGS) (Figure 2), available for download via Pharos Digital library, can be used to generate corrections to the reported moment from the MPMS 3. These corrections can be applied any time after the measurment is complete through post-processing the data. This tool allows users to correct for both sample size/shape effects and radial offsets. The reported moment for any sample that differs in size/shape from the QD calibration standard (palladium cylinder, 2.8 mm in diameter and 3.8 mm in height) will suffer some degree of error.

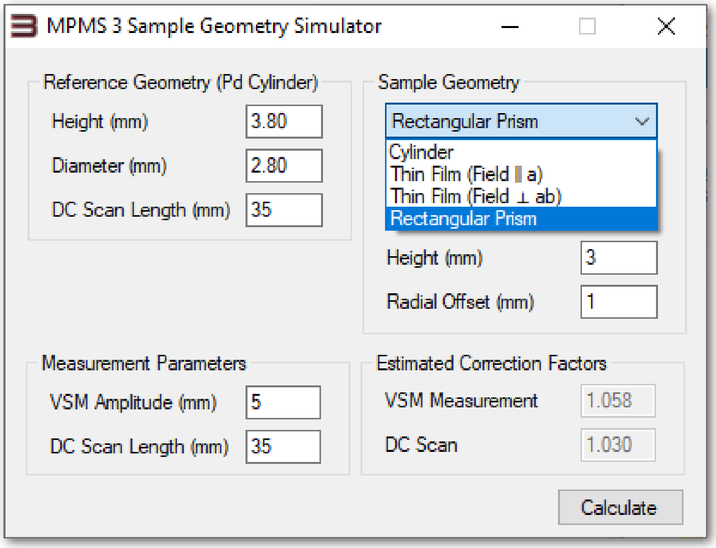

Figure 2: Sample geometry simulator for the MPMS 3. Note, one must ***divide*** *the measured moment by the appropriate Estimated Correction Factor.*

QD Palladium (Pd) Standard

The palladium reference sample, shown in Figure 3, is chosen as the calibration standard for all QD magnetometers, including the legacy MPMS and PPMS VSM/ACMSII measurement options. The high purity (3N5) Pd samples are supplied by Princeton Scientific. Prior comparisons (Note 1041-001) to the NIST standard SRM765 show a typical discrepancy of less than 0.5%. The Pd reference included for each MPMS 3 system is a laser cut cylinder, 2.8 mm in diameter and 3.8 mm in height, with a nominal mass of ~240 mg. Purity is further verified by measuring the low field moment vs. field response to confirm that there is a minimal nonlinear component.

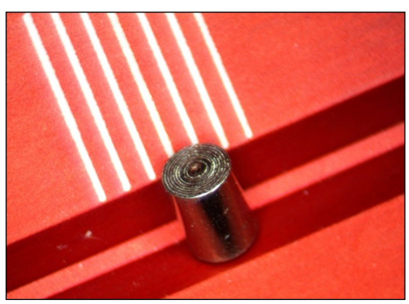

Figure 3: Palladium reference sample.

Once an MPMS 3 system is properly calibrated, the measurement of the Pd should yield constant moment values for different vibration amplitudes and scan lengths, using either the SQUID-VSM or the DC-scan measurement modes, respectively. The Pd standard is mounted in a quartz tube holder with a dab of GE7031 varnish. After longitudinally centering the sample using the standard installation Wizard a rotational test is performed to identify the minimum moment position in order to position the sample as close as possible to the radial center. As shown in Figure 4, the moment (T = 298 K and H = 1 T) variation extracted from SQUID-VSM (1-8 mm) and DC-scan (30-60 mm) measurements is less than 0.2%.

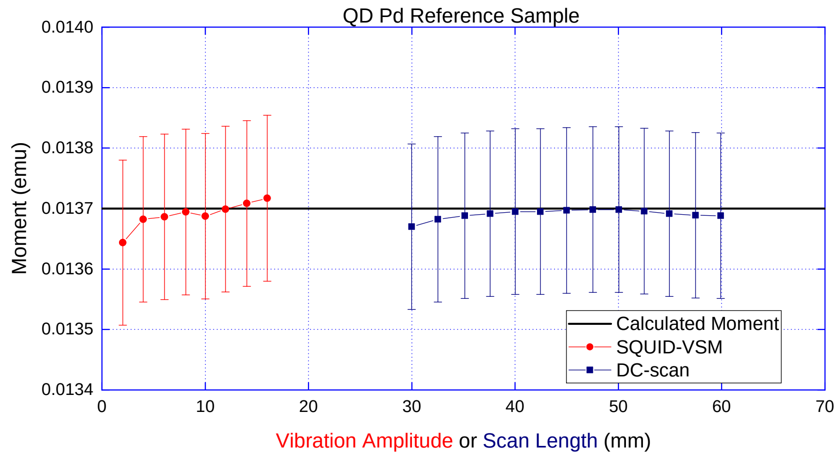

Figure 4: The calibrated and measured Pd sample moment is independent of vibration amplitude (SQUID-VSM) and scan length (DC-scan). All reported measurements are within 1% (the instrumental uncertainty, shown via error bars) of the calculated value based on the Pd sample's mass and the applied field.

Factory calibrations ensure the measured moment extracted from SQUID-VSM and DC-scan measurements are not only consistent with each other but also do not depend on the vibration amplitude nor scan length. This calibration is only strictly true for samples with the same size/shape as the Pd sample. In the following we will examine two additional samples, a rectangular prism and a small sphere, to demonstrate the reported moment dependence on scan length and how to correct the measured moment using the SGS.

QD Er:YAG Sample

The QD Er:YAG (Princeton Scientific, 20 at% Er doping) sample shown in Figure 5 was a prior[4] reference standard for AC magnetometers used in both MPMS and PPMS platforms. The shape of the sample is a rectangular prism with dimensions 3x3x2 mm^3.

[4] It has since been replaced by a GGG cylinder with the same size/shape as the Pd sample.

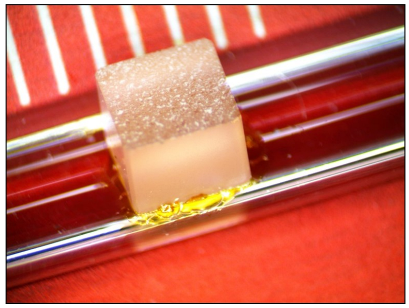

Figure 5: Er:YAG reference sample (now obsolete).

The Er:YAG reference is mounted on a quartz paddle sample holder, which would nominally introduce a 1 mm radial offset, Figure 5. After centering the sample, the moment is measured at T = 300 K and H = 1 T using both SQUID-VSM and DC-scan modes, Figure 6. Interestingly, the measured SQUID-VSM moment shows a very strong dependence on the vibration amplitude (solid red circles), while DC-scan moment values (solid blue squares)are constant over a wide range of scan lengths. As the Er:YAG sample has a very different shape compared to the Pd standard such differences are not unexpected.

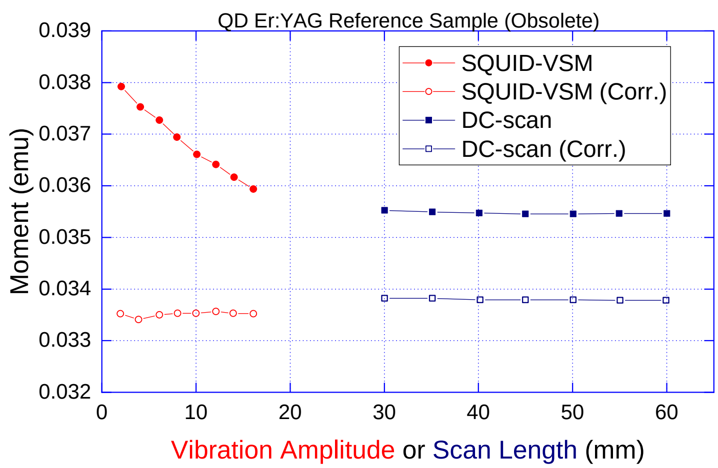

Figure 6: After implementing the correction factor from the SGS, the Er:YAG sample moment is independent of vibration amplitude and scan length (open symbols).

Correction factors were estimated using the SGS with the following input parameters:

- Geometry: Rectangular Prism
- Width (mm): 3
- Depth (mm): 3
- Height (mm): 2
- Radial Offset (mm): 0.65

Note, the radial offset is based on a best guess. It is a variable that can be tuned to optimize the correction. As shown in Figure 6, after applying the correction (by ***dividing*** by the provided Estimated

Correction Factor provided by the SGC) the moment variation between SQUID-VSM (open red circles) and DC-scan (open blue squares) measurements is less than 1%.

NIST YIG Magnetic Standard (SRM 2853)

A yttrium iron garnet (YIG, SRM 2853) sphere (1 mm diameter, ~2.8 mg), Figure 7, is measured using the same procedure.

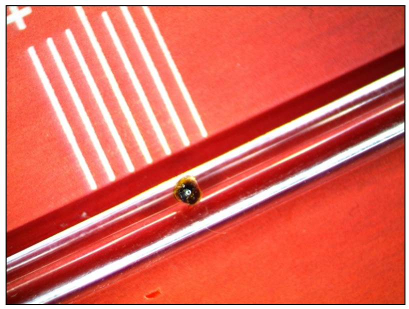

Figure 7: NIST YIG standard.

The measured moment is shown in Figure 8. Similar to the aforementioned Er:YAG sample, the SQUID-VSM moment shows a strong dependence on vibration amplitude (solid red circles) while the DC-scan moment values show no clear dependence on the scan length (solid blue squares).

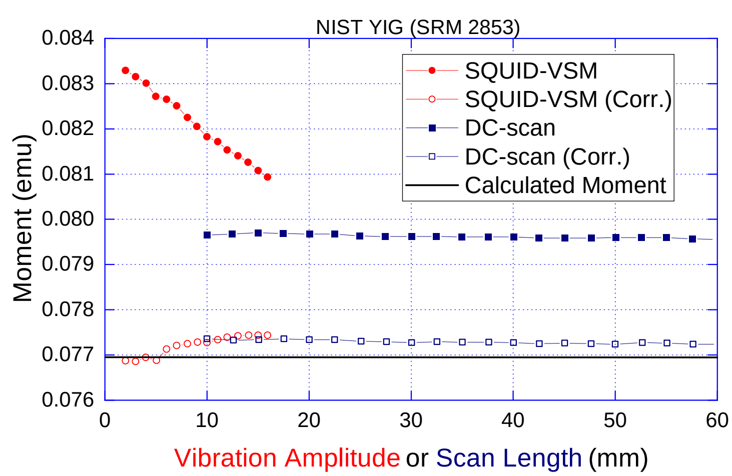

Figure 8: After implementing the correction factor from the SGS, the YIG sphere sample moment is independent of vibration amplitude and scan length (open symbols).

Correction factors were then estimated using the SGS with the following parameters:

- Geometry: Cylinder
- Diameter (mm): 0
- Height (mm): 0
- Radial Offset (mm): 0.5

As a small homogeneously magnetized sphere can be approximated as a point source, a cylinder with zero volume is used. As shown in Figure 8, after applying the correction the moment variation between SQUID-VSM (open red circles) and DC-scan measurements (open blue squares) is less than 1%. Furthermore the corrected MPMS 3 moment values agree with the NIST value to within 0.6%.

Summary

The measured magnetic moment of samples which differ in size and/or shape as compared to the Pd reference sample, or are radially offset within the gradiometer, will suffer some degree of accuracy loss. We have shown, with different references and standards, that it is possible to approach the "true" moment of test samples to within 1% in the MPMS 3 systems using the SGS. The radial offset is the most difficult quantity to account for. A recent publication **[4]** shows a unique technique to improve measurement accuracy by measuring both SQUID-VSM and DC-scans.

References

[1] R.K. Dumas, T. Hogan. "*Recent Advances in SQUID Magnetometry*" Magnetic Measurement Techniques for Materials Characterization, edited by V. Franco and B. Dodrill, Springer International Publishing, 2021; DOI.

[2] M. Coak, C. Liu, D.M. Jarvis, S. Park, M.J. Cliffe, and P.A. Goddard, SquidlLab-A user-friendly program for background subtraction and fitting of magnetization data, Review of Scientific Instruments, 91, 023901 (2020); DOI.

[3] P. Stamenov and J.M.D. Coey, *Sample size, position, and structure effects on magnetization measurements using second-order gradiometer pickup coils*, Review of Scientific Instruments, 77, 015106 (2006); DOI.

[4] C.O. Amorim, F. Mohseni, R.K. Dumas, V.S. Amaral, and J.S. Amaral, *A geometry-independent moment correction method for the MPMS3 SQUID-based magnetometer*, Measurement Science and Technology, 32, 105602 (2021); DOI.