MPMS 3 Application Note 1500-020

Accuracy of Reported Sample Moment: Using the Sample Geometry Simulator

Abstract

This Note discuss the accuracy of the sample moment reported by MPMS 3, common factors that introduce moment offsets, and examples of making moment correction using the MPMS 3 Sample Geometry Simulator which is available on the Pharos library at https://www.qdusa.com/pharos/view.php?fDocumentId=1734.

Moment Accuracy

The accuracy of the reported sample moment has always been of great importance for MPMS users, especially when the moment values are used as inputs for further quantitative analysis, such as a Curie-Weiss law fit to the susceptibility vs. temperature data. Also, some users tried to compare the moment values obtained from different QD magnetometers, such as MPMS XL, MPMS 3, and PPMS VSM, using the "same" sample and found unexplained discrepancies.

MPMS is a highly automated SQUID magnetometer with sophisticated hardware and software, capable of generating quality data at impressive speed. On the other hand, just like any other experimental measurement apparatus, it does not readily output the "true" moment values. Depending on how knowledgeable and diligent the operator is with the measurement process, the difference between the reported and the "true" moment can range from less than 1% to larger than 10%.

The measurement process of MPMS has been thoroughly discussed in previous QD Application Notes, posted at www.qdusa.com (1014-214; 1014-822). In simple terms, it involves moving a sample though the second order gradiometer thus inducing a position dependent voltage waveform, which is in turn fitted to a theoretical response curve. The magnetic moment value of the sample is one of the fitting parameters. A few key factors affect the accuracy of the reported moment to varying degree, including:

- Sample mounting (App Notes 1014-201, 1096-306)
- Background of sample holder (App Note 1014-213)
- Sample centering, longitudinal and radial (App Note 1500-010)
- Sample shape (App Note 1500-015)
- Magnetic Field accuracy (App Notes 1500-011)
- Demagnetization effects (see 1992 writeup from R. Goldfarb at NIST here)

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 only relevant in materials in regions of high susceptibility $\chi = dM/dH$ (ferromagnets and superconductors). Of highest importance are centering accuracy and sample shape which can have significant effects on moment error. Centering matters in both longitudinal and radial directions (Figure 1). The longitudinal centering is performed automatically by the system. However, when sample signal is small, for example, a weak magnetic thin film on a substrate, user judgment is required to correctly center the sample. Radial centering on the other hand is less obvious and often overlooked, yet it has a large impact on moment error, especially when sample dimensions are much smaller compared to the diameter of the sample chamber (~8 mm). A quick way for checking radial centering is to rotate the sample rod and take measurements at a few angular positions (Figure 1, right). A large variation with angle indicates strong radial offset. The position with *minimum* moment value is likely to be closest to the center of the sample chamber.

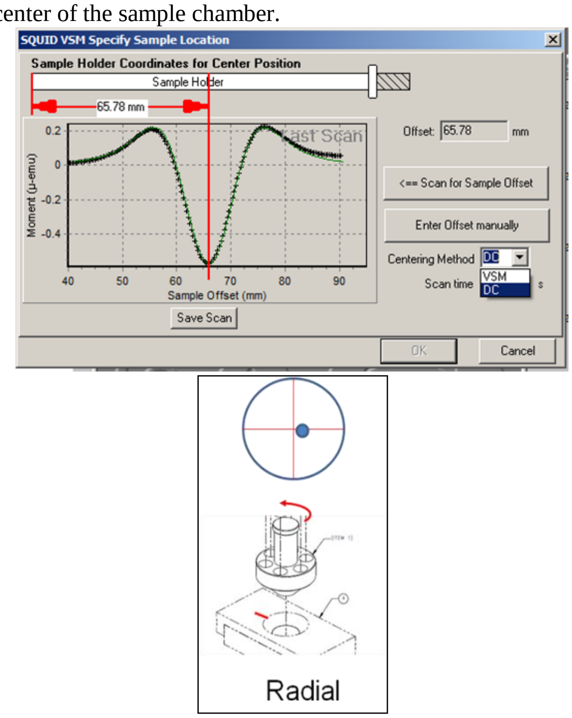

Figure 1. Sample centering: Longitudinal and Radial

For post measurement correction in MPMS 3 we now offer the Sample Geometry Simulator (SGS) (Figure 2, download available via QD Pharos Digital library). It allows users to correct for both, radial offset and sample shape effects. Sample shape error arises when the shape of the test sample is different from the QD calibration standard, the Palladium cylinder, 2.8 mm in diameter and 3.8 mm in height.

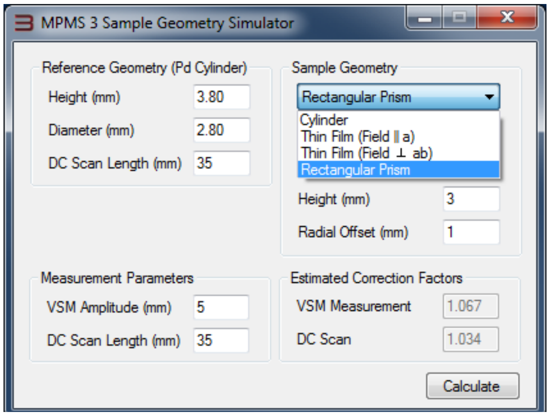

Figure 2. Sample Geometry Simulator for MPMS 3

QD Palladium (Pd) Standard

Signal output of the magnetometers are voltage waveforms. To convert that into a moment value one needs a reference sample. The Palladium reference sample shown in Figure 3 is chosen as the calibration standard for all QD magnetometers (DC), including MPMS and PPMS VSM. The high purity (3N5) Pd samples are supplied by Princeton Scientific. Previous tests (App Note 1041-001) made comparison to NIST standard SRM765 and found error to be less than 0.5%. The Pd reference furnished for each MPMS 3 system is 2.8 mm in diameter and 3.8 mm in height. Nominal mass is around 240 mg with slight variation from system to system. To avoid magnetic contamination, all Pd samples were laser cut to the exact dimensions. Purity is further verified using low field M vs. H in the SQUID system to confirm that there is minimal nonlinear component.

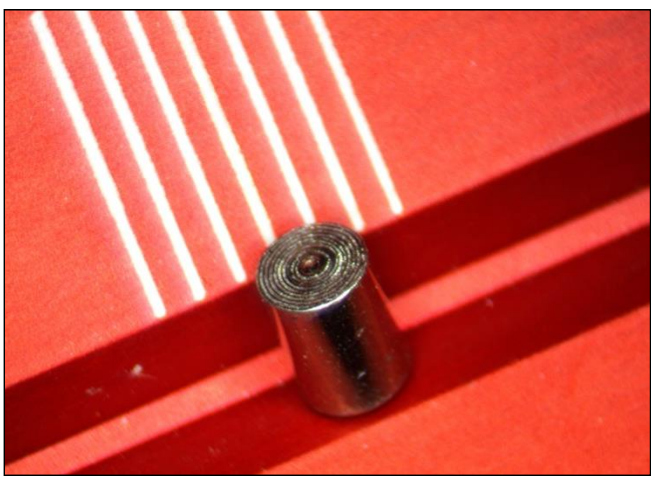

Figure 3. QD magnetic moment reference, Palladium

Once an MPMS 3 system is properly calibrated, the measurement of Pd should yield essentially constant moment value for different scan length, in either the VSM or the DC Scan measurement modes. For MPMS 3, the Pd standard is mounted in a quartz tube holder with a tiny dab of GE varnish. After centering the sample using the Change Sample 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. Then set T = 298 K and H = 1 Tesla, and acquire data from 1 mm to 8 mm amplitude in VSM mode, and 30 mm to 60 mm scan length in DC Scan mode. As shown in Figure 4, the moment variation over scan length is less than 0.2% from 2 mm to 60 mm.

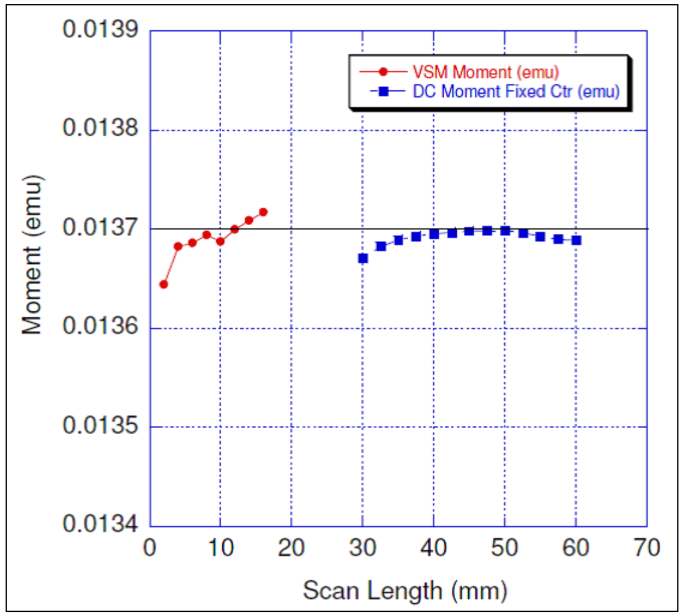

Figure 4. Pd sample moment independent vs scan length

Amplitude correction is part of the system final test procedure to make sure results from VSM and DC Scan measurements are consistent. This scan length independence does not remain true however, if the test sample has a different shape than the Pd reference. 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 for it using the SGS.

QD Er:YAG Sample

The QD Er:YAG sample shown in Figure 5 is the reference standard for AC magnetometers in MPMS and PPMS. Also supplied by Princeton Scientific, the laser grade high purity YAG crystal comes with 20at% Er doping. The Er:YAG sample is a paramagnetic insulator, so the AC out of phase component is always zero over the entire frequency range. The shape of the sample is rectangular prism with dimensions 3x3x2 mm^3.

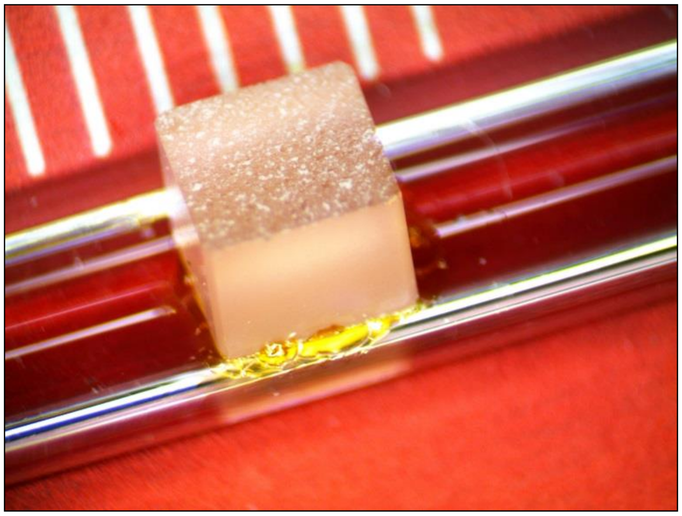

Figure 5. Er:YAG sample: Rectangular Prism

The Er:YAG sample is mounted on a quartz paddle sample holder. After centering the sample, set T = 300 K and H = 1 Tesla, then acquire data from 1 mm to 8 mm amplitude in VSM mode, and 30 mm to 60 mm scan length in DC Scan mode. Shown in Figure 6, VSM moment vs. scan length shows a strong dependence (open circles), while DC Scan moment values are fairly constant from 30 mm to 60 mm (triangles). The dependence is due to the fact that Er:YAG sample has a very different shape compared to the Pd standard.

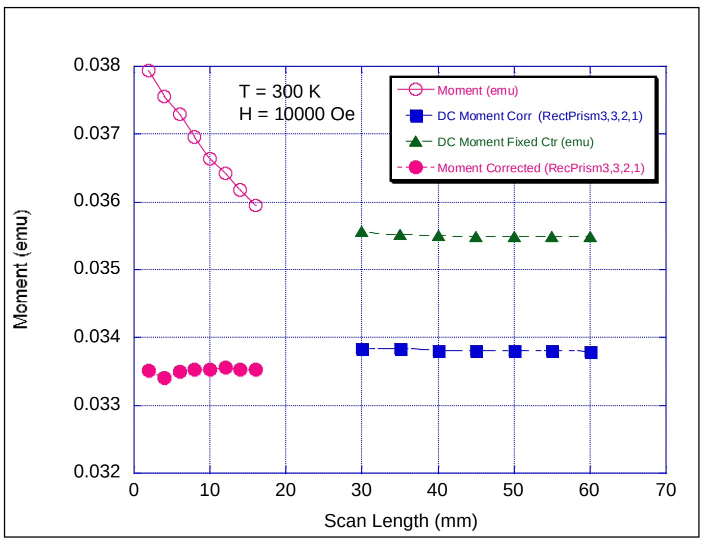

Figure 6. Er:YAG sample moment independent vs scan length

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

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

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 the moment variation between VSM moment (solid circles) and DC Scan moment (squares) is less than 1%.

NIST YIG Magnetic Standard SRM 2853

Next we tested NIST magnetic moment standard, Yttrium Iron Garnet (YIG, SRM 2853) following the same procedure. The YIG standard is a sphere with nominal diameter of 1 mm and nominal mass of 2.8 mg, see Figure 7.

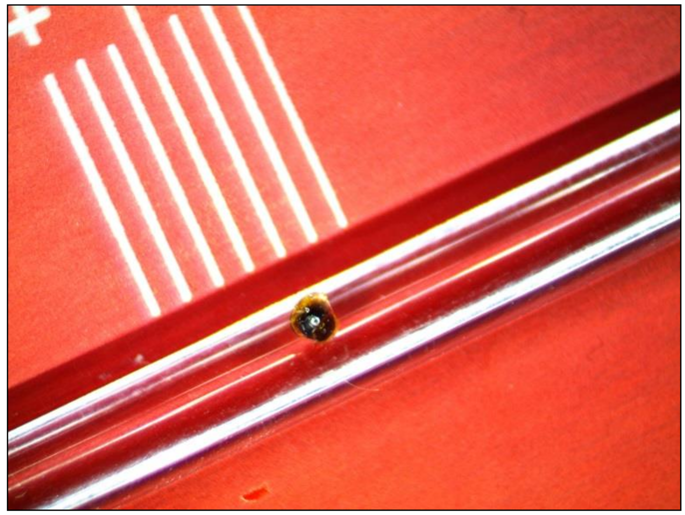

Figure 7. Picture of NIST YIG standard on Quartz sample holder

The YIG sphere is mounted on a quartz paddle holder on the mid line. After centering the sample, set T = 300 K and H = 0.5 Tesla, then acquire data from 1 mm to 8 mm amplitude in VSM mode, and 30 mm to 60 mm scan length in DC Scan mode. Shown in Figure 8, the VSM moment vs. scan length shows a strong dependence (blue circles), while DC Scan moment values are fairly constant from 30 mm to 60 mm (blue triangles).

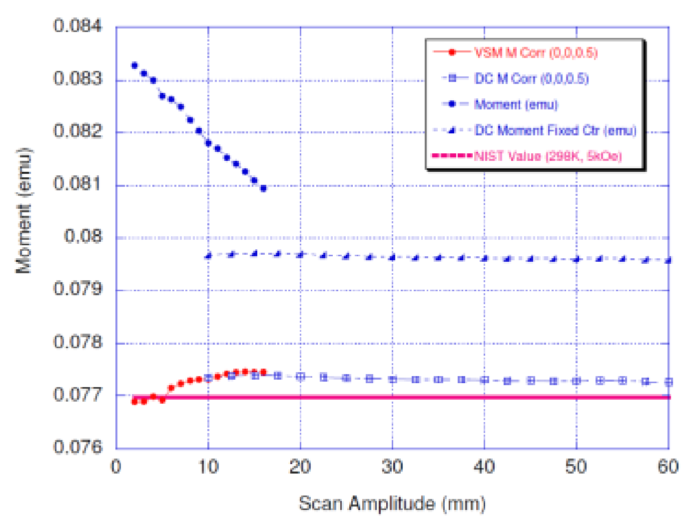

Figure 8. YIG sample moment independent vs scan length

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

Sample Geometry: Cylinder

Diameter (mm): 0

Height (mm): 0

Radial Offset (mm): 0.5

As a homogeneously magnetized sphere is magnetically identical to a point source, we used a cylinder with zero dimensions. As shown in Figure 8, after applying the correction the moment variation between VSM moment (red circles) and DC Scan moment (blue squares) is less than 1%. MPMS 3 reported moment values (corrected) agree with the NIST value to within 0.6%.

Summary

We have shown, with different NIST standards that it is possible to approach the "true" moment of test samples to within 1% in the MPMS 3 systems. However, effort is needed on the operator side to optimize sample mounting, measurement process, and post measurement corrections using the SGS.