

DC Moment: Measured Two Ways

Measurements of the field and/or temperature dependence of the DC magnetic moment are a necessary, and often first, step when studying magnetic materials. Inductive measurement techniques, whether they utilize a traditional vibrating sample magnetometer (VSM) or superconducting quantum interference device (SQUID) are ubiquitous and provide a quantitative measure of the magnetic moment. The SQUID-based magnetic property measurement system (MPMS[®]3) from Quantum Design [1] uses two complementary measurement protocols to calculate the same quantity, namely the DC magnetic moment. This Application Note aims to first describe the differences between the traditional DC-Scan and SQUID-VSM modes and under what circumstances one should use each. Then, by using both DC-Scan and SQUID-VSM measurements, a geometry-independent scale factor is demonstrated to improve measurement accuracy.

DC-Scan

The traditional DC-Scan measurement mode, summarized in Figure 1, relies on moving a magnetic sample through the entirety of a superconducting 2nd-order gradiometer. Currents generated in the gradiometer are inductively coupled to the SQUID which acts as a sensitive current-to-voltage transducer. The position dependent voltage waveform, $V(z)$, is then fit using the functional form shown in Figure 1 to calculate the magnetic moment, after suitable calibrations are applied. The accuracy of the resulting magnetic moment is directly linked to the quality of this fit.

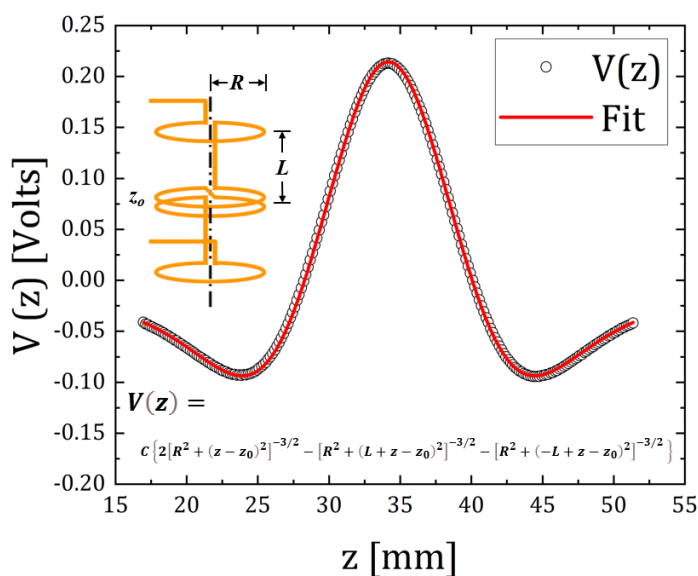


Figure 1: DC-Scan basics. Typical voltage waveform, $V(z)$, and 2nd-order gradiometer.

SQUID-VSM

For the SQUID-VSM mode, summarized in **Figure 2**, the sample oscillates sinusoidally at the center of the 2nd-order gradiometer with a given frequency (ω) and amplitude (A). As the spatially-dependent voltage waveform is parabolic near the center of the gradiometer, $V(z) = z^2$, this results in a time-dependent voltage, $V(t)$, at twice the physical oscillation frequency. Standard lock-in techniques can be used to detect this now time-dependent voltage. The in-phase voltage amplitude is then used to calculate the DC magnetic moment, after suitable calibrations are applied.

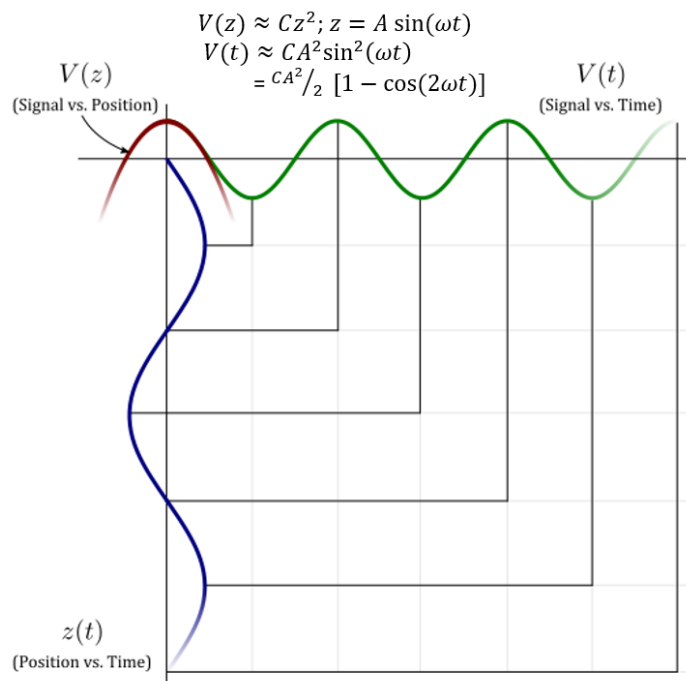


Figure 2: SQUID-VSM basics. Schematic highlighting how the position dependent voltage waveform $V(z)$ transforms into a time-dependent $V(t)$ at twice the oscillation frequency.

When to Use the DC-Scan Mode

The tried-and-true DC-Scan, which has been utilized since the early 1980's, is still relevant in several situations. The slow and gradual motion employed during a DC-Scan measurement is better suited for certain sample mounting techniques and sample holders. For example, liquid samples generally benefit from DC-Scan measurements as the liquid will undoubtedly jostle too much when vibrated. Furthermore, vibrating a large and relatively massive sample holder, e.g. pressure cell, generally results in temperature instabilities and are best measured with slow DC-Scans. Finally, as the DC-Scan mode naturally measures the response as a function of position, ensuring the sample is properly centered is straightforward. This is much more difficult for SQUID-VSM measurements where it is a time-dependent voltage that is being measured, and additional calibrations need to be carried out to ensure the sample remains centered during variable temperature measurements.

Benefits of the SQUID-VSM Mode

The SQUID-VSM mode was developed due to the numerous benefits over the traditional DC-Scan mode, these include:

Sensitivity and Dynamic Range

The sensitivity of the SQUID-VSM mode is approximately a factor of 5 better than the DC-Scan mode. This is not surprising given the additional lock-in detection required. Furthermore, due to the strong dependence of measured voltage on the amplitude, $V(t) \sim A^2$, the dynamic range, spanning 10 orders of magnitude, is significantly larger for the SQUID-VSM mode. A large vibration amplitude is best for low-moment samples to maximize the generated voltage, and small amplitudes can be used to measure large-moments without saturating the sensitive SQUID detection circuitry.

Uniform Temperature/Field Profile

Typical DC-Scans require movement of the sample over distances of at least 35 mm, whereas the largest SQUID-VSM amplitudes are 8 mm. For all else equal, the sample will always be in a more uniform temperature and field environment for a SQUID-VSM measurement as compared to a typical DC-Scan measurement. This is usually not critical except for samples, e.g. superconductors, where small changes in temperature and field can have dramatic effects on their magnetic properties.

Measurement Speed

Data acquisition is generally an order of magnitude faster for the SQUID-VSM mode. This results in either a faster turnaround between sample measurements or a significant increase in point density. This can be an important time saver if one wants to precisely measure the temperature dependence of a sharp $m(T)$ phase transition or acquire as many points as possible near a sharp $m(H)$ switching field.

Insensitive to SQUID Drift

The SQUID voltage naturally slowly drifts with time. Remember, the absolute SQUID voltage is generally not critical, only the differences observed upon translating or oscillating the sample through the gradiometer. If this drift is linear, as it usually is over short time scales, it can be easily accounted for and subtracted from the DC-Scan $V(z)$ waveform. However, DC-Scans are particularly sensitive to nonlinear SQUID drift. Nonlinear SQUID drift can arise from multiple sources, but the most prevalent is changing the external magnetic field in unidirectional steps, as one would be required to perform for a standard $m(H)$ hysteresis loop measurement. For details related to the superconducting solenoid used to generate the applied field, this nonlinear SQUID drift is also most prevalent for magnetic field magnitudes less than 7500 Oe. As nonlinear SQUID drift cannot be properly accounted for during a DC-Scan, measurements are usually far noisier than their counterparts measured using the SQUID-VSM mode (see [Figure 3](#) for exemplary hysteresis loops).

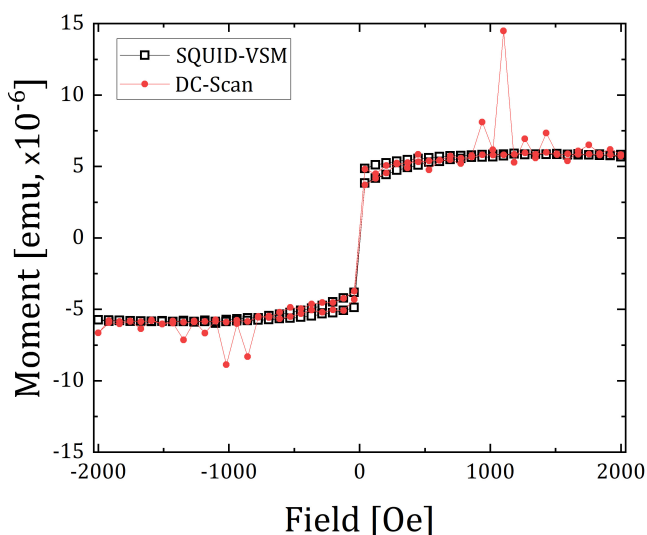


Figure 3: Comparison of the DC-Scan (red circles) and SQUID-VSM (black squares).

Easier Background Subtraction

Depending on the sample holder and sample mounting, a measurement of the background moment may be required. The process is different depending on if the measurements were obtained via a DC-Scan or the SQUID-VSM mode.

As the DC-Scan requires first fitting a position-dependent voltage waveform to calculate the resulting magnetic moment, one cannot simply subtract the background moment ($m_{\text{background}}$) from the sample+background moment ($m_{\text{sample+background}}$). Instead, it is recommended to first subtract the corresponding voltage waveforms from each other: $V(z)_{\text{background+sample}} - V(z)_{\text{background}}$ and then refit the resulting sample voltage waveform to calculate the magnetic moment. Application Note 1500-023 describes more about this procedure [2]. Additionally, there are freely available software packages that help facilitate this process [3].

However, for measurements using the SQUID-VSM mode one can simply subtract the measured moment of the background from that of the sample+background measurement: $m_{\text{background+sample}} - m_{\text{background}}$. Therefore, for low-moment samples and/or sample holders with non-negligible backgrounds, the SQUID-VSM mode would be much preferred and expected to yield far more accurate results compared to the DC-Scan mode.

DC-Scan + SQUID-VSM: Calculate a Geometry-Independent Scale Factor

The SQUID-based MPMS 3 is one of the most sensitive commercial magnetometers on the market. In addition to sensitivity, accuracy is also often of paramount importance for most researchers. **A point often ignored is that the accuracy of the reported moment depends on the size and shape of the sample being measured as compared to the sample used to calibrate the instrument.** If the sample size and/or shape differs significantly from the calibration sample, a significant loss (>10%)

of accuracy is not uncommon. For the MPMS 3, a Pd cylinder (3.8 mm height/2.8 mm diameter) is used as the calibration standard. It's also important to remember that even for this calibration standard, our accuracy specification is to within $\pm 1\%$. Furthermore, one of the most often overlooked factors is the radial centering of the sample within the gradiometer. The radial centering is difficult to estimate and adjust. Most importantly, radial centering can be the most significant factor in determining measurement accuracy.

As it is often impossible to match the size/shape of the test sample to the calibration sample, corrections often need to be performed in post-processing, which usually assumes well-defined sample geometries and a well-defined radial offset [4]. Such ideal conditions can limit the accuracy of the correction factors and calculated moment.

A recent publication [5] empirically uncovered a systematic relation between the normalized difference, x , of SQUID-VSM and DC-Scan measurements:

$$x = (m_{\text{SQUID-VSM}} - m_{\text{DC-Scan}}) / m_{\text{SQUID-VSM}}$$

It turns out for a given DC-Scan length and SQUID-VSM vibration amplitude this difference, x , can be modeled by a simple 3rd-order polynomial:

$$\alpha(x) = 1 + Ax + Bx^2 + Cx^3$$

Finally, the corrected moment can be calculated as:

$$m_{\text{Corrected}} = m_{\text{SQUID-VSM}} / \alpha(x)$$

Interestingly, this relation follows a clear and predictive trend, independent of sample geometry, i.e. size and shape, and radial offset for a given pair of DC-Scan and SQUID-VSM measurements. Exploiting this trend, a geometry-independent correction can be calculated by simply measuring the DC moment two different ways.

This technique is demonstrated using a 0.025 mm thick Ni foil (Alfa Aesar, 99.5%) cut into a 7 mm \times 4 mm sheet that is glued to a quartz paddle sample holder, **Figure 4** (inset). Clearly this sample geometry differs significantly from the Pd reference sample. At saturation, the measured moment for the SQUID-VSM (black squares) and DC-Scan (red circles) modes differ from one another by approximately 11% ($x = -0.11$). This difference is expected and consistent with the fact that the sample is not of the same size/shape as the Pd reference. The empirically determined polynomial coefficients for a SQUID-VSM amplitude of 5 mm and DC-Scan length of 35 mm are, $A = 2.068$, $B = 4.00$, $C = 7.1$ [6], resulting in a correction factor of $\alpha(-0.11) = 0.808$. Dividing the as-measured SQUID-VSM moment by this factor yields the corrected moment (blue triangles), which agrees exceptionally well (within 1%) with the expected moment calculated from the sample mass (3.2 mg) and magnetization [7].

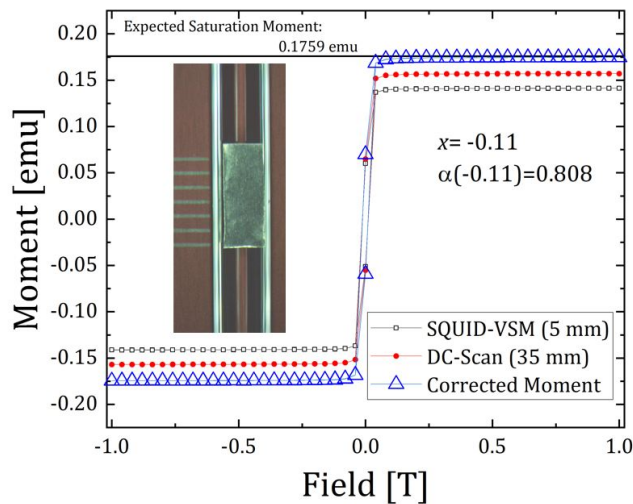


Figure 4: DC moment of a thin Ni foil measured with the SQUID-VSM (black squares) and DC-Scan (red circles) modes. The corrected moment (blue triangles) agrees with the expected saturation moment.

To further test the robustness of this geometry-independent scale factor calculation, the same Ni foil is folded into a thin strip, **Figure 5** (inset). As the exact same mass of Ni is being measured, the saturation moment should remain unchanged. Interestingly, two differences are observed for this altered sample geometry. Firstly, the measured moment for the DC-Scan and SQUID-VSM modes are now larger than the expected moment. Secondly, the SQUID-VSM moment is now larger than the DC-Scan moment. These differences are a direct consequence of the sample geometry. The difference between DC-Scan and SQUID-VSM measurements is now much smaller, about 2.7%. Nevertheless, the calculated scale factor, $\alpha = 1.06$, results in a corrected moment that is within 1% of the expected value. Further examples of this geometry independent scale factor are presented in Reference [8].

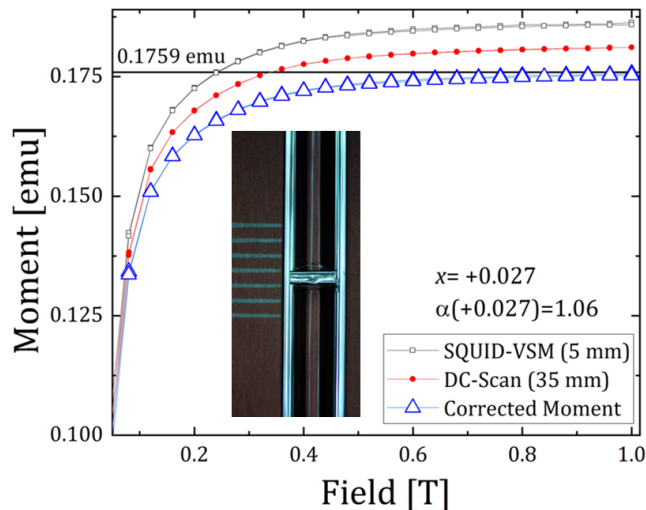


Figure 5: DC moment of the same Ni foil used in **Figure 4**, but now folded into a thin strip. The measured moment using the SQUID-VSM (black squares) and DC-Scan (red circles) modes now show the opposite trend as observed in **Figure 4**, yet the corrected moment (blue triangles) agrees with the expected saturation moment as the total mass has been preserved.

Summary

The SQUID-based MPMS 3 from Quantum Design is a versatile DC and AC magnetometer that enables sensitive sample measurements over a wide temperature (1.8-400 K for the base system) and magnetic field (± 7 T) range. The DC magnetic moment, in particular the differences and utility of the DC-Scan and SQUID-VSM detection modes, was the primary focus for this Application Note. While it may at first seem redundant to have two techniques that measure the same fundamental property, each detection mode has its own unique benefits depending on the sample and sample mounting constraints. Finally, by measuring the DC magnetic moment with both the DC-Scan and SQUID-VSM modes one can calculate a scaling factor that is independent of the sample size, shape, and radial offset that can be used to dramatically improve the accuracy of the measured DC moment. This technique is particularly useful for samples that are irregularly shaped and/or cannot be modified to mimic the size/shape of the Pd reference sample and where measurement accuracy is of prime importance.

References

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- [6] See reference [5].
- [7] NIST Standard Reference Material SRS 762. <https://tsapps.nist.gov/srmext/certificates/762.pdf>
- [8] See reference [5].