Quantum Design

Application Note 1014-824

Measuring Large Moment Samples in the Magnetic Property Measurement System (MPMS)

This note explains the best procedures for achieving reliable data when measuring samples with magnetic moments up to the instrument specification of ±5 emu. To summarize, a 64-point DC scan should be used when the sample moment exceeds 1.0 emu. Special procedures also are required when the RSO transport is used for automated measurement of sample moments over 0.1 emu. One solution is to add the “Measure DC” sequence command directly after each “Measure RSO” sequence command to produce a second data set. Quantum Design also offers the Extended Dynamic Range (EDR) option, which is used to measure moments in excess of 300 emu.

General Background

Quantum Design provides two standard transport options to be used with the MPMS. The DC transport is part of the base system and performs the stepped DC scan. The DC transport and its slide-seal assembly are required for a number of measurement applications, such as the Sample Rotator or the Fiberoptic Sample Holder (FOSH). The RSO transport is primarily used for samples with small magnetic moments. The RSO transport is preferred for routine magnetic measurements because it can perform both the stepped DC scan technique and the synchronous RSO scan technique. In addition, the synchronous RSO scan technique offers various styles of sample motion.

The DC and RSO measurement techniques provide complementary means for achieving the large dynamic range of the MPMS. The strength of the RSO technique is that it offers a relatively fast type of measurement, which then allows more averaging and better noise rejection than the DC technique. While these features improve the achievable sensitivity of the MPMS, they place limits on the determination of large moments. Using the RSO technique, the standard 4 cm Center Scan cycle will only report values less than 0.4 emu. A 4 mm Center Scan can report values up to 5.0 emu, but with loss of accuracy. The Max Slope technique has a detection limit of 2.5 emu for a 3 mm amplitude. Furthermore, extreme care must be used in centering samples that will be used for short scan-length measurements. Otherwise, the data will be misinterpreted by the fitting algorithm.

The MPMS moves the sample through a second-order gradiometer superconducting pickup loop. At the beginning of each scan, the SQUID detector is reset to near zero (0) volts. If the SQUID voltage exceeds the limit of the hardware (±10 V for DC; ±5 V for RSO), then the system will perform an auto-range. An increase in the range and gain will increase the scale of the detection system. The scale can be increased up to the maximum setting of 1.25 emu scale on a standard instrument (see *MPMS Application Note 1014-213*, "Subtracting the Sample Holder Background from Dilute Samples" for a description of this process).

With the RSO technique, if the SQUID returns a value larger than ±5 V while on the maximum scale, an overrange error is reported to the log file and no data is recorded in the data file. By contrast, the DC measurement technique will always report a value to the data file. At maximum scale, the DC technique will repeatedly reset the SQUID during a scan once specific conditions have been met. With the DC technique, the fundamental limitation is the ±10 V per step size. Thus, a 4 cm 64-point scan will reliably measure moments in excess of the specification of ±5.0 emu. Note that it takes significantly longer to perform a 64-point DC scan with the RSO transport than it does with the DC transport.

DC Technique

The DC technique starts at the lowest point of the scan length and sequentially moves the sample upward through the superconducting pickup loops, at each step reporting the SQUID voltage to the "DATA.DC.RAW" data file. The sample is returned to the starting point at the end of each scan. A user should typically select a 4 cm, 32-point scan. The primary SQUID calibration factor is determined by using the palladium reference standard and a 4 cm, 24-point scan. However, at least 32 points are required to achieve the specification of better than 1% accuracy for moments within ±5.0 emu.

When the moment is in excess of 1.0 emu, underestimation of the moment increases as the number of points decreases. This relation is illustrated in Figure 1, which shows the raw SQUID voltage versus scan distance for a sample of powdered erbium oxide with a moment of 9.4 emu. Both the 24-point and 64-point scans produce a voltage exceeding ±10 V, reflecting the additional measurement requirement of resetting the SQUID during the scan.

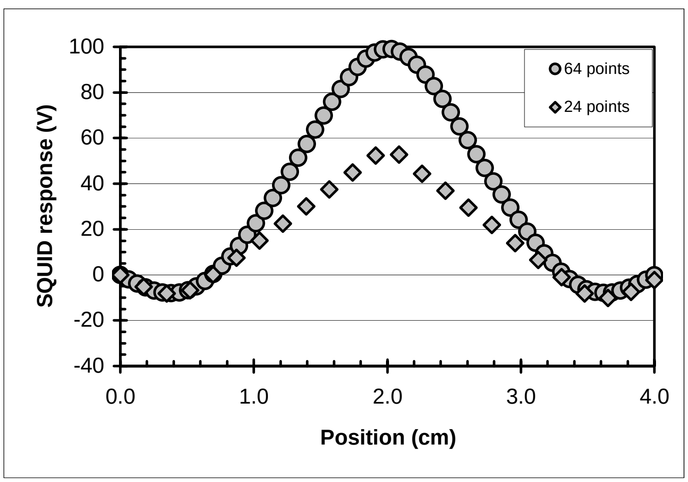

Figure 1. The response function is shown for a sample of Er_2O_3 with a moment of 9.4 emu at 10 K and 3 tesla. The data are the raw SQUID voltages, with the circles representing 64-point scans and the diamonds representing 24-point scans.

By comparison, the much smaller amplitude for the 24-point scan is the direct result of the larger step size. The larger step size causes the change in voltage per step to exceed ±10 V in the critical maximum slope regions of the response curve.

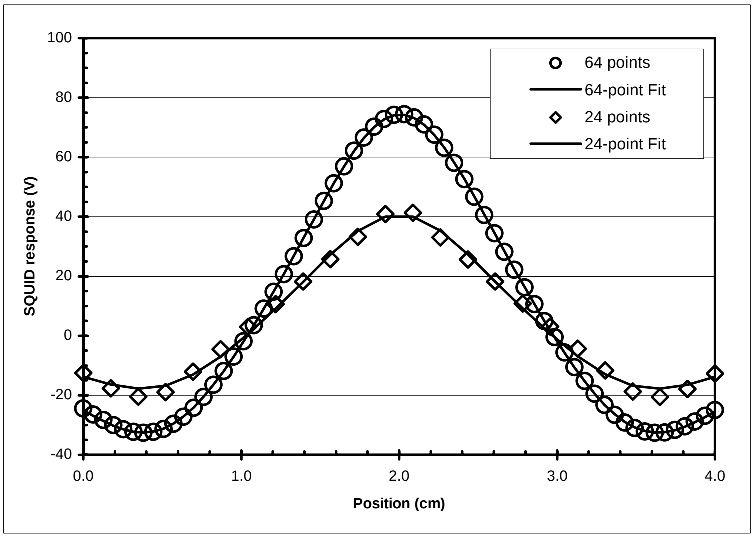

Figure 2. The longitudinal detrended data and iterative regression fits for the response functions are shown in Figure 1. The open symbols are the longitudinal detrended data; the line is a longitudinal detrended fit to the data. The circles and diamonds represent 64-point scans and 24-point scans.

In Figure 2, the data from Figure 1 have been detrended and a longitudinal detrended fit has been applied using the iterative regression algorithm. The poor quality of fit for the 24-point scan is readily apparent, as the response function does not correlate to a point-source dipole.

Figure 3 shows the reported value of the moment for the erbium oxide sample at room temperature as a function of the applied field and number of points per scan. The correct values of the moment were determined by performing a 4 cm, 32-point DC scan using an instrument equipped with extended dynamic range (EDR). The figure shows an obvious correlation between the saturated reported value of the moment and the number of points per scan. In addition, the underestimation of the true value of the moment begins well before the saturated moment value has been reached.

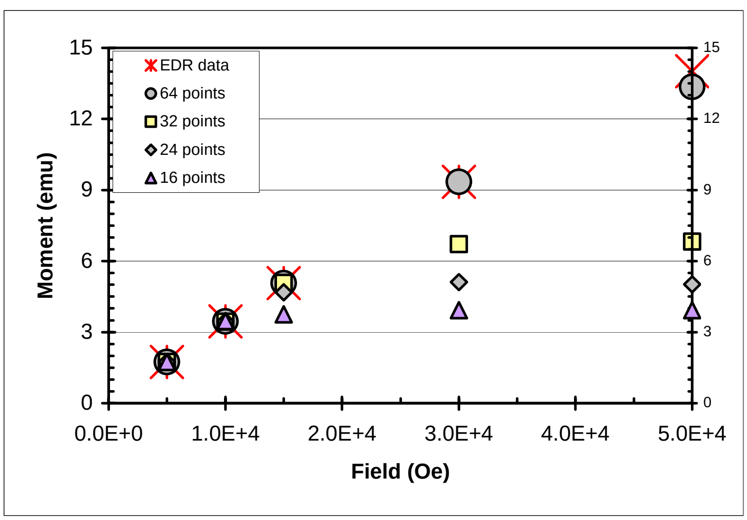

Figure 3. Magnetization versus field data for an Er_2O_3 sample at 10 K shows the dependence of the reported moment value on the number of steps used in the DC scan. Comparison is made to the true value as obtained by using the extended dynamic range (EDR) option.

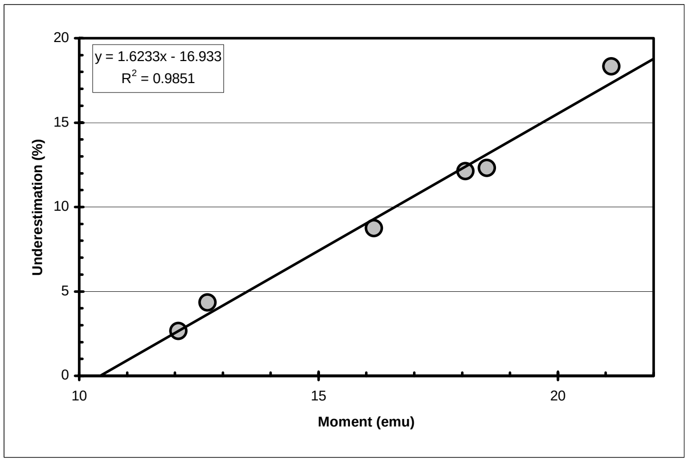

Figure 4. The percentage underestimation of the moment (obtained without using EDR) is plotted versus the true moment value (obtained by using EDR). The formula shows a linear relation between the magnitude of the moment and the percentage of underestimation.

The percentage of underestimation is predictable, as illustrated in Figure 4. Below 10.0 emu, the value reported with EDR is equivalent to data collected without EDR for a 4 cm, 64-point scan. However, as the moment increases beyond 10.0 emu, there is a gradual increase in the extent of underestimation. The percentage underestimation is obtained using equation (1):

$$\text{Underestimation (\%)} = 100 * (\text{true value} - \text{reported value}) / \text{true value}. \quad (1)$$

The true value is determined by using an EDR-enabled system and a 4 cm, 64-point scan. There is roughly a linear correlation between the percentage of underestimation and the size of the moment. This correlation extends to at least twice the value of the last reliable measurement.

The upper limit of the DC technique can be increased beyond 10.0 emu by decreasing the scan length. However, you must use extreme care when you center the sample. For short scan lengths, the longitudinal voltage and longitudinal detrended voltage should be identical. To examine the raw data, display the longitudinal voltage on the y2 axis. If the sample is off center, the response-curve analysis software will assume there is a large SQUID drift and will subtract a linear contribution from the raw data. Ultimately, the software will report a value for the moment that is substantially lower than the true value.

RSO Center Scan Technique

The RSO Center Scan technique starts the sample in the middle of the scan length and proceeds to move the sample down, then up, then back down to stop in the center. If this is the only measurement, the sample will remain in the center position during subsequent temperature or field changes. The 64 discrete points of average SQUID voltage per cycle versus sample position are recorded in the “DATA.RSO.RAW” data file. If the SQUID returns a value larger than ±5 V while on the maximum 1.25 emu scale, an overrange error is reported to the log file and no information is recorded in any of the data files. The maximum change in voltage in the response function must be less than 5 volts for scan lengths of 4 cm or longer. This change in voltage corresponds to roughly 0.4 emu.

To exceed 0.4 emu with a Center Scan, the scan length must be shortened so that the change in volts is less than 5 volts. A reasonable lower limit for the Center Scan length is about 4 mm, where the slight curvature near the exact center of the gradiometer is used to estimate the moment. While this technique can record moments up to 5.0 emu, the data will not be very accurate.

When short scan lengths are used, the longitudinal voltage and longitudinal detrended voltage should be identical. To examine the raw data, display the longitudinal voltage on the y2 axis. If the sample is off center, the response-curve analysis software will subtract a linear contribution from the raw data, on the assumption that there is a large SQUID drift. Ultimately, the software will report a value for the moment that is substantially lower than the true value.

RSO Max Slope Technique

The Max Slope technique starts from a predefined center position. The sample is then moved up 0.62 cm to be in the middle of the calculated region of maximum slope. Measurements that are performed with Max Slope commonly have smaller amplitudes and higher frequencies relative to the RSO Center Scan technique. After the Max Slope measurement command, the sample will remain in the Max Slope region during subsequent temperature or field changes. The 64 discrete points of average SQUID voltage per cycle versus sample position are recorded in the "DATA.RSO.RAW" data file. If the SQUID returns a value larger than ±5 V while on the maximum 1.25 emu scale, an overrange error is reported to the log file and no information is recorded in any of the data files.

The Max Slope technique is commonly used when taking very fast measurements during magnetization versus field [M(H)] loops. Another appropriate use of the Max Slope technique is to minimize the effect of field inhomogeneity when measuring with very small amplitudes. The process uses the entire 10 V range of the RSO electronics. Thus, a typical 5 mm scan length can measure moments up to 1.0 emu. Because a linear-fit algorithm is applied to Max Slope raw data, any absolute positional error will result in a substantial loss of accuracy.

Extended Dynamic Range

The scale of the MPMS can be greatly increased by addition of the Extended Dynamic Range (EDR) option. To obtain this option on an existing probe, return it to Quantum Design. Here the EDR hardware will be incorporated into the SQUID probe assembly on the temperature control module (TCM).

The EDR system is activated automatically when the scale is 1.25 emu and the critical voltage has been exceeded. In EDR mode, current is supplied to a resistor located in series between the pickup loop and SQUID detector, which modifies by a constant amount the voltage seen by the SQUID. The calibration of this constant must be checked after the EDR-enabled probe has been installed. The EDR option is active when the emu scale reads a multiple of the calibration factor rather than the standard values. When the MPMS is working at the highest scale setting with an active EDR option, all the software features and measurement considerations described for maximum scale measurements apply.