Application Note 1500-018

Using Straw Sample Holder with DC Scan Mode

Abstract

This Note introduces hardware and software features of the new DC Scan mode in MPMS 3 system, and offers useful guidance on using straw sample holders.

Introduction

In addition to quartz and brass sample holders, DC Scan mode in MPMS 3 also enables researchers to use translucent plastic straws for measurement. This note provides a brief introduction to the DC Scan mode, hardware needed for using straws, and software features. Sample mounting and measurement parameters are also discussed. Researchers are ultimately responsible for assuring optimal outcome of the measurements based on adequate understanding of the experimental process and conditions.

It is strongly recommended, that new users of DC Scan review MPMS application note **Sample Mounting Consideration 1014-201** (Quantum Design, 2000) for basic understanding of sample mounting issues in MPMS. The Note offers many important tips for using straw sample holder that are fully applicable to DC Scan measurements in MPMS 3.

DC Scan Mode

In principle, the DC Scan in MPMS 3 works the same as the Standard Scan in MPMS XL and other earlier MPMS models. The DC scan technique involves moving a sample linearly over several centimeters through second-order gradiometer detection coils, which induces a SQUID voltage as a function of sample position curve (Figure 1). Magnetic moment of the sample is derived from fitting analysis of the induced voltage curve to a single dipole response function.

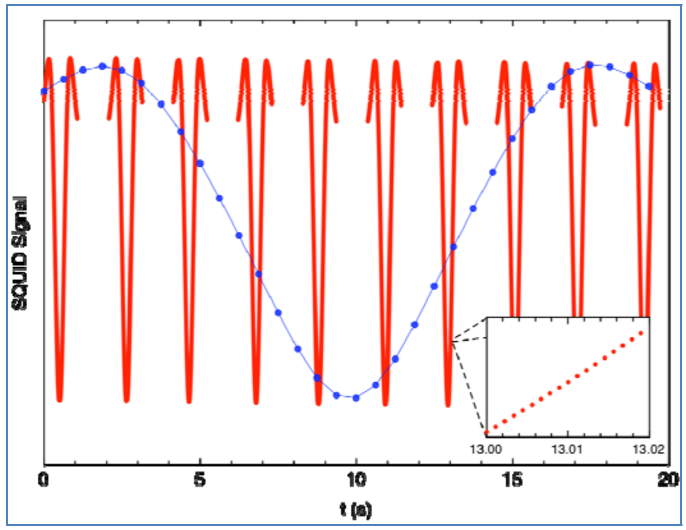

Figure 1. Ten MPMS 3 DC waveforms (red) generated in 20 seconds, compared to one XL standard waveform (blue)

Technically the DC Scan is greatly improved thanks to the latest hardware and software developments. In MPMS 3, continuous linear sample motion at constant speed is realized with the help of a high precision motor with position resolution down to 10 micron. Due to the new architecture of the measurement platform, sample position data and SQUID voltage are independently gathered, then recombined synchronously as a function of time. Each DC Scan curve now contains over 600 data points, compared to the 60 data points in an XL scan (Figure 1). The DC Scan speed is also 10 times faster than the Standard Scan in XL.

The DC Scan is also better designed in dealing with the SQUID voltage drift. Unlike the Standard Scan, where drift is removed from the raw voltage as a linear term in the fitting function, each measurement in DC Scan includes an UP scan and a DOWN scan, so the SQUID drift is first removed in time space before the fitting analysis is performed in position space. This new method is not sensitive to slight vertical centering error of the sample, nor the asymmetric signal introduced by end effects.

DC Scan Mode

The hardware needed for using a straw sample holder include: the straw adaptor (QD part # **4500-614**) and straws (QD part # **8000-001**). As shown in Figure 2, the straw fits snugly onto the barb of the adaptor, and sample is mounted securely in the straw. A sample mounting station, which is part of the standard MPMS 3 user kit, should be used to position the sample in the straw around the "66 mm" mark. More tips and guidelines are provided in a later section on sample mounting. Please contact your QD representative if you need assistance getting these parts.

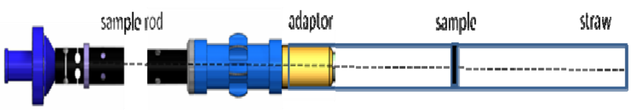

Figure 2. Sample rod, straw adaptor(yellow), and straw holder with sample

Note: Straw length should be cut so that it is roughly the same as the quartz or brass sample holders.

Caution: Certain types of (Non-QD) straws may melt above 350 K!

Software Version and Features

For using DC Scan, your system needs to have MultiVu version **2.0** or newer. Please contact your QD representative if you need assistance with the software upgrade.

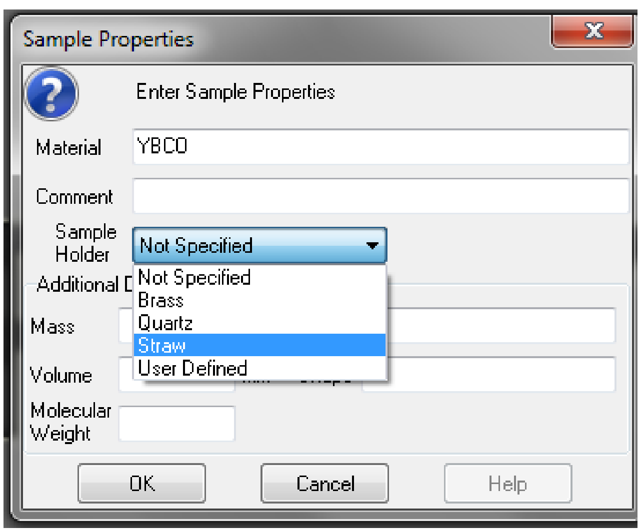

Figure 3. Sample Properties panel for selecting sample holder type

Shown in Figure 3 is the Sample Properties panel, where the user enters sample information and selects sample holder type when installing a new sample into the MPMS 3 system. It is important to remember to choose "Straw" when you are using straw sample holders. The importance of this is related to an advanced feature called Auto-Tracking, where the system automatically tracks the center of the sample during cooling and warming, based on predetermined thermal expansion properties of the sample rod-sample holder assembly. In the case of "Straw" selection, the parameters are preset based on the type of straw provided by QD. If you choose straws from other sources, it is recommended to use clear plastic straws with minimal magnetic signal. When using straws from sources other than QD one should also expect different behavior of sample position upon cooling and warming.

Figure 4 shows the Measurement panel where users can set DC scan parameters, including Scan Length, Scans per Meas., Scan Time, and a check for saving Raw Data.

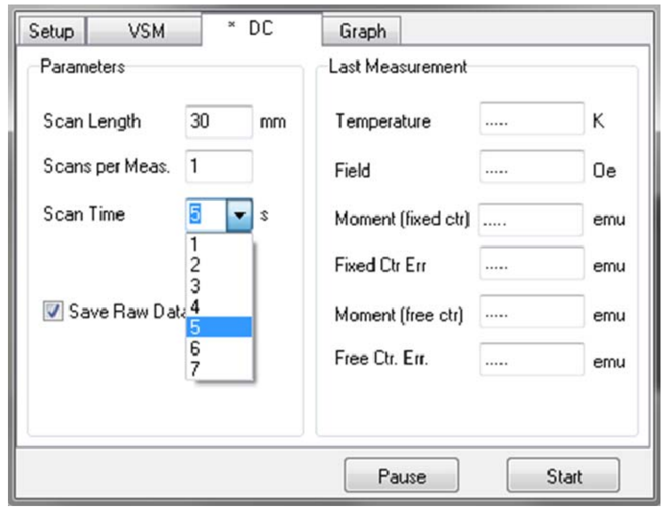

Figure 4. Measurement panel for setting DC Scan parameters

Scan Length can be set from 10 mm to 60 mm. In general, 30 mm should be used as the minimum scan length. A scan length smaller than 30 mm can lead to inaccurate reported moment (see later section on measurement and data for more discussion). In special cases, a shorter scan length might be used to reduce heat shuttling effect inside the sample chamber, especially at very low temperatures. But its applicability needs to be carefully examined, and the reported moment corrected accordingly.

At scan lengths longer than 30 mm, the potential issue would be temperature instability due to the large sample movement. Another point to keep in mind is field non-uniformity over the scan length, which could generate considerable artifacts in particular temperature or field regimes for a given sample. For further reading on the impact of field non-uniformity please refer to **Effects of Field Uniformity on the Measurement of Superconducting Samples** (Quantum Design, 1998).

The number of Scans per Measurement is typically set to 1 or 2. More scans per measurement can help increase signal to noise, a very useful technique when running M(H) sequence on samples with small moment.

Scan Time can range from 1 s to 7 s, with 1 s being the fastest scan speed. In general, for larger moment values, slower scans should be used to avoid software errors in reconstruction of DC waveforms. For some measurements (e.g., time dependence of magnetization), a faster scan would be more appropriate. At the same time, users need to be mindful that faster scans could generate more frictional heat, as well as exerting a larger force on the sample. Typically, 3s or 4s could be good starting points for experiment. Total run time saved by using faster scan is insignificant compared to the run time shortened by the QuickSwitch™ magnet technology in MPMS 3.

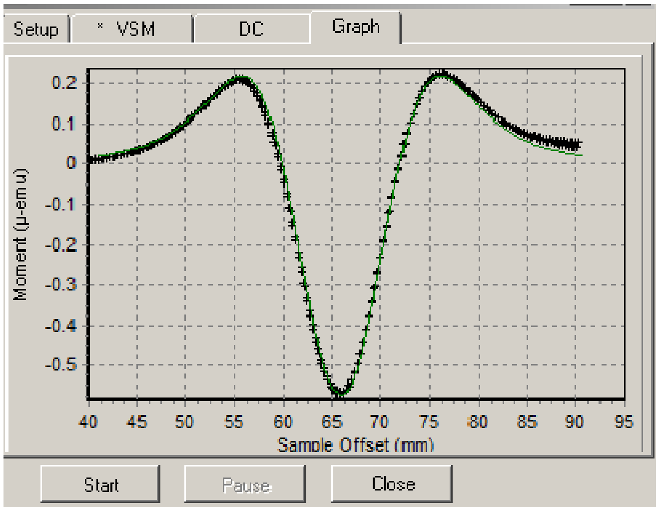

Figure 5. Measurement panel Graph tab showing raw voltage curve of last scan

Figure 5 shows the raw voltage waveforms (UP and DOWN scans) of last measurement (black curve) and the mathematical fit (green) to the raw waveform. MPMS 3 DC Scan allows users to save all raw voltage for review and post processing. If the "Save Raw Data" is checked, software will produce both a “xx.dat” file and a “xx.raw” file for the measurement sequence executed. It is important to point out that, for the reported moment value to be meaningful, the raw waveform needs to closely resemble the single dipole response curve, like the waveform shown in Figure 5.

To graph DC Scan data please choose **DC Moment Fixed Ctr (emu)** or **DC Moment Free Ctr (emu)** in Graph panel (see Figure 6). MPMS 3 offers both the Fixed Center fit moment, and the Free Center fit moment. These are equivalent to the **Linear Reg. Fit** and **Iterative Reg. Fit** in XL. For Fixed Ctr fit, the central peak of the fitting curve is always fixed at the center of the scan, while amplitude is varied to best match the raw data. For Free Ctr. fit, the central peak of the fitting curve is shifted around to minimize the fitting error; it tends to hunt for the largest peak in the raw voltage curve, and sometimes leads to sudden sign changes in reported moment. The goodness of the fitting, named **DC Fixed Fit** or **DC Free Fit**, should be a number at least between **0.9** to **1**, with **1** being the perfect fit. It's a good habit to always check this number, and review the raw voltage curves to validate the measurement results.

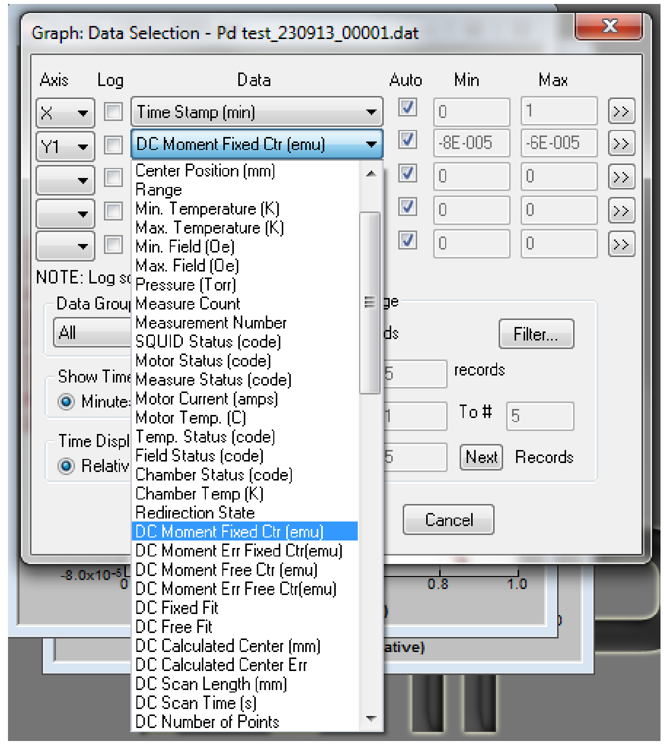

Figure 6.Data Graph panel showing DC Scan related data columns

Sample Mounting

In principle MPMS 3 is detecting local permeability changes inside the 2^{nd} order gradiometer detection coils. This means a small point sample will induce a perfect single dipole response, while a long uniform line-shape sample that extends beyond the gradiometer baseline of ~ 2 cm does not produce a signal. This forms the basic guideline for sample mounting: minimize background contribution from materials that are used to secure or support the sample, including varnish, straw pieces, plastic disks, crossed threads, etc. Below we provide a few "good practice" examples of using straw sample holders.

1. Uniform background material

Use two short sections of straws with thru cuts, and slide them into the outer straw to sandwich the sample in the middle. Their near identical (try your best to make it so) shape and length make sure the background is uniform, and thus contribute little to the overall signal.

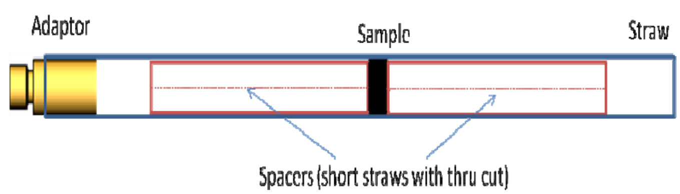

Figure 7a. Sample mounting in straw

2. No background material

For thin film samples, one can make the sample size (~ 5x5 mm) such that the thin film can be wedged into the straw snuggly. This works for both field parallel and perpendicular to the thin film substrate. Cotton Q-tips can be inserted from both ends of the straw to manipulate the thin film sample into its desired position.

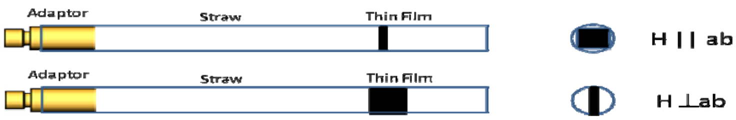

Figure 7b. Thin film sample mounting in straw

3. Minimal background material

A cross of white cotton threads is the lowest mass background platform for mounting a sample, and is especially useful for single crystals. Use a clean, non-magnetic needle to thread some white cotton thread through the straw walls and tie a secure knot so the thread cross is rigid. A tiny dab of varnish or grease can be used to secure the sample on the thread cross.

Figure 7c. Single crystal sample mounted on crossed thread platform

Gelatin or polycarbonate capsules (QD part No. **AGC3**) are both low background platforms ideal for powder samples. It is important to contain powder samples so the sample chamber is not contaminated. One method is to embed powder in a low susceptibility epoxy like Duco cement. It's good to keep the powder near the center of the capsule, or completely fill the capsule (Figure 7).

Figure 7d. Powder sample in gelatin capsule.

Users are advised to find more details regarding sample mounting materials and methods in the following application notes:

- MPMS 3 application note **VSM Sample Mounting Techniques (1096-306)**, Quantum Design, 2010.
- MPMS application note **Sample Mounting Considerations (1014-201)**, Quantum Design, 2000.

Measurement and Data

MPMS 3 DC Scan is calibrated using the Pd standard sample from Quantum Design, the same standard used in all magnetic measurement platforms including MPMS XL and PPMS VSM. More information on the Pd standard can be found in application note **Palladium Reference Sample (1041-001)**, Quantum Design, 2005.

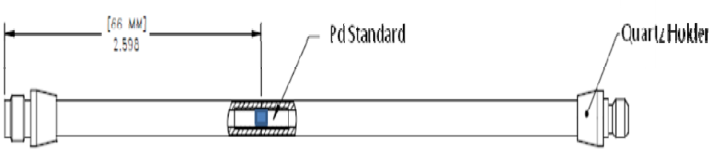

Figure 8. MPMS 3 Pd standard assembly

As shown in Figure 9, both DC Scan mode and VSM mode are calibrated using the Pd standard so that they agree perfectly, and at the same time, matching the result from MPMS XL (blue curves in Figure 9).

The frequently asked question is: when is the reported moment the "true moment" of the sample? If your sample has the same shape as the Pd standard (cylinder, L = 3.8 mm, D = 2.8 mm), AND, if it is perfectly centered in the same quartz sample holder like the Pd sample, the moment measured then is accurate and true. If, for example, the sample is a small sphere, then there will be an error offset from the "true moment" value. As shown by the red curves in Figure 9, the measured moment of a YIG sphere (NIST magnetic standard 2853) in both MPMS 3 and XL, while showing perfect agreement with each other, are both off by about 2% from the "true" moment of the NIST reference sample.

This error, which is easily corrected by a factor independent of temperature or field changes, is due to the geometric shape difference of your sample and the Pd standard, thus the different ways they interact with the 2nd order gradiometer detection coils. Please find more discussions on how to correct for sample shape error in **Accuracy of the Reported Moment: Sample Shape Effects (1500-015)**, Quantum Design, 2010.

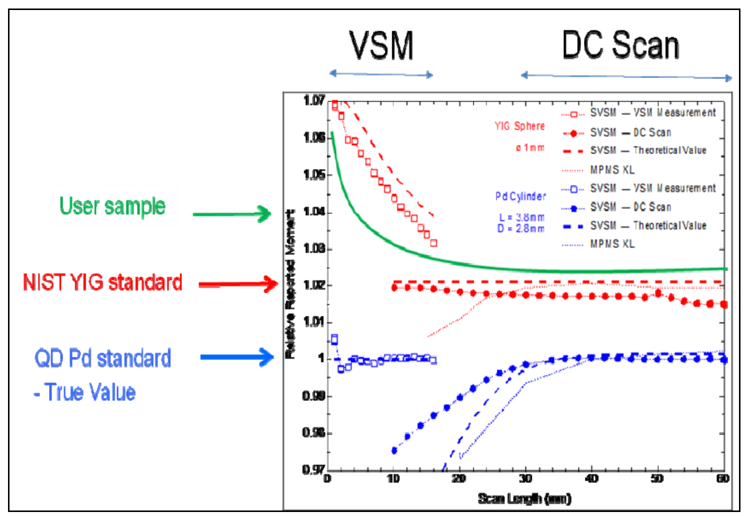

Figure 9. Normalized moment of Pd standard and NIST standard

Another source of moment error can arise from radial positioning of the sample. In other words, the sample is not perfectly centered radially. This is typically a smaller error compared to the geometric shape effect. One can easily test moment variation with angle, in a particular mounting method, by rotating the sample rod from the top of the sample transport and taking measurements manually at several angles. For in-depth discussions please see the application note **Accuracy of the Reported Moment: Axial and Radial Sample Positioning Error (1500-010)**, Quantum Design, 2010.

When the raw waveform does not look like a single dipole response [see Figures 10(a) and (b)], the curve fitting and reported moment are no longer meaningful, in which case background subtraction becomes necessary. In DC Scan mode, the correct method for background subtraction is to first subtract the raw V(z) waveform of sample plus background from V(z) of background alone, then perform curve fitting of the resultant curve[Figure 10(c)], which should resemble the symmetric single dipole response waveform.

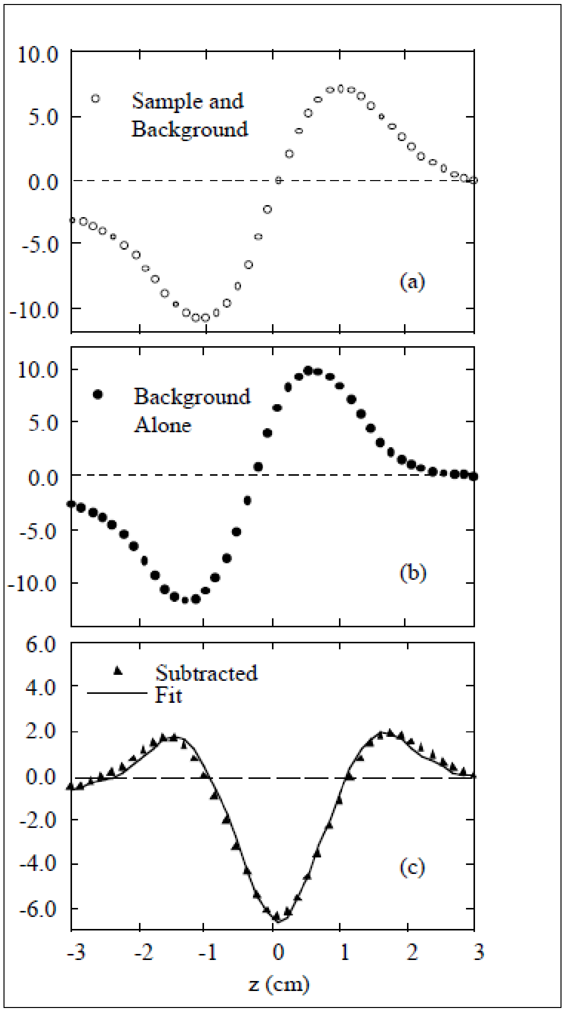

Figure 10. Background subtraction in DC Scan mode

The automated background subtraction routine for MPMS 3 will be featured in a future software release.