

Application Note 1500-026

Calibrating the Auto-Tracking capabilities

for a custom sample holder using the MPMS3

Background

The initial installation and centering of a sample in the MPMS3 occurs at room temperature. However, as the measurement temperature is varied the position of the sample with respect to the center of the 2nd order gradiometer will vary due to differential expansion/contraction of the materials comprising the sample holder and sample chamber. The amount of thermal expansion/contraction is typically on the order of a few millimeters over the approximately 400 K temperature range of the MPMS3. Therefore, when performing a measurement as a function of temperature it is always recommended to enable *Auto-Tracking*, especially when measuring using the SQUID-VSM detection mode. The *Auto-Tracking* feature will automatically adjust the center position with changing temperature to keep the sample centered in the gradiometer. The *Brass*, *Quartz*, *Straw*, *Rotator*, and *Magneto Optic (FOSH)* sample holders have all been pre-calibrated at the factory. The user also has a single *User Defined* sample holder option for custom sample holders, *e.g.* a pressure cell. This Application Note provides a step-by-step guide on performing the *Auto-Tracking* calibration for the *User Defined* sample holder.

Step 1: Download the AutoTracking Calibration.bas script file from Pharos:

The <u>AutoTracking Calibration.bas</u> file can be found in our online digital database *Pharos* within the following directory:

Customer Area/MPMS3/Measurement App Notes/AutoTracking Calibration

Save this file to the *QDSquidVsm/Macros* directory on the MPMS3 computer.

It is best to be running the latest version of MultiVu, release 2.3.4.15 or later. To upgrade to the latest version of MultiVu please contact your <u>local service representative</u>

Step 2: Prepare your custom sample holder:

Ideally the custom sample holder length should mimic total length of one of the standard (*e.g.* brass or quartz) holders, with the sample position approximately 66 mm from the bottom of the sample holder. Remember, the sample chamber has a diameter of 9 mm. During the calibration process the magnetic moment will be measured and the location tracked over a $400 \leftarrow 4$ K

temperature range. Therefore, it is important that the test sample has an easily measurable magnetic moment over the entire temperature range and the material construction of the custom sample holder should also be able to withstand these temperatures.

Load a small and highly magnetic test sample inside the custom sample holder; for example, a small piece of Fe (the size of the Pd reference standard or smaller) would be a good test sample. Ensure that the maximum moment of the test sample is no more than 2 emu at the chosen measurement field.

Attach your custom sample holder onto a sample rod and load into the MPMS3 using the *Sample Installation Wizard*, as you would with any other sample. Once the sample is centered, complete the *Wizard* such that the sample chamber is in a *Purged* state.

Step 3: Run the AutoTracking Calibration.bas script:

Under the Sequence menu near the top of the MultiVu window select Advanced \rightarrow Run Macro and double-click on the AutoTracking Calibration.bas file located in the QdSquidVsm/Macros directory where it was saved earlier. This will immediately bring up the following window:

medsarement octup	1921
Sample Holder	Custom
Sample:	Pd
Field (Oe):	10000

Figure 1. Auto Tracking Calibration measurement parameters.

At this stage a name can be given to the *Sample Holder* to identify it, the magnetic test *Sample* can be identified, and the measurement *Field (Oe)* specified. The measurement temperature range spanning 400 K $\leftarrow \rightarrow$ 4 K is preset within the *.bas* file.

Click OK and the script will set the specified measurement *Field (Oe)* and measurement temperature to 400 K. Once stable, the temperature will first ramp to 4 K and then back to 400 K, continuously measuring the moment using the DC scan mode. The script will take approximately 150 minutes to complete and return the sample chamber to room temperature and the magnetic field to 0 Oe. Once finished the following plot will appear showing the measurement results:



Figure 2. Auto-Tracking Calibration measurement data.

The corresponding files are saved in the *QdSquidVsm/Data/AutoTracking* directory:

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Figure 3. Location of the Auto-Tracking Calibration measurements.

The most important file generated is the *.ini* file, which upon opening contains a single line which quantifies the *Auto-Tracking* parameters to be used by the MPMS3:



Figure 4. Auto Tracking .ini file containing the calibration data.

Copy this information to the *clipboard* using *ctrl-c*.

Step 4: Update the vsm.ini file:

The *vsm.ini* file can be found in QdSquidVsm/SVsm/System:

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JIII Installation	1.1.16.347	10/19/2016 11:05	File folder
Lab SQUID	J.1.16.372	10/19/2016 11:05	File folder
Service Firmware	DCScanProcess.dll	10/20/2017 2:34 PM	DLL File
	seqtrans_vsm_0.dll	10/20/2017 2:34 PM	DLL File
Macros	SVsmMultiVu.dll	2/7/2018 9:49 AM	DLL File
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Figure 5. Location of the vsm.ini file.

Open the *vsm.ini* file and *Paste (ctrl-v)* the single line generated during after completion of the *AutoTracking Calibration.bas* script as shown below:

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File Edit Format View Help	
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OVEN TYPE=1 AUTOTRACK:USER DEFINED=6.638e-6 ; Custom (20180223-163819)	

Figure 6. Paste the Auto-Tracking Calibration data into the vsm.ini file.

Save/overwrite the now modified vsm.ini file.

It is then necessary to **Restart** *MultiVu* for these changes to take place.

Note: *MultiVu* can only accept one *User Defined* sample holder. Therefore, if multiple entries for different sample holders are entered into the *vsm.ini* file, the entries not to be used must be commented out using a semi-colon, (;).

Note: Whenever modifications to the *vsm.ini* file are saved, MultiVu must be restarted for those changes to take place.

For questions, contact apps@qdusa.com