



MPMS Application Note 1014-822

Verifying Absolute Sensitivity in the MPMS

This application note presents the sample characteristics and measurement sequence used to verify the absolute sensitivity of the Magnetic Property Measurement System. The procedure provides data during initial installation and routine inspection of basic system parameters.

The general concept consists of mounting a small amount of well-defined paramagnetic material on a low background sample holder. At high fields, a sufficiently large dc signal permits the sample to be properly centered. A small applied field allows evaluation of the system's absolute sensitivity. A moment of approximately 1×10^{-8} emu at room temperature is expected in a field of 10 Oe.

Preparing the Sample

Mount a sample of about 2-20 μg of Er_2O_3 permanently in quartz. Perform this synthesis in a fume hood while wearing proper eye protection to safeguard your eyes from the intense UV light source when the quartz is heated.

Obtain 20cm of 3mm-diameter solid quartz rod (GM Associates). Place a small amount of erbium oxide (Alfa Aesar, 99.99% purity) powder at the 10cm midpoint on the rod. Use a hydrogen/oxygen torch to diffuse the sample into the quartz. Gently apply heat to the region until the quartz is glowing and the green fluorescence from the erbium is visible. Try not to bend the quartz rod while heating. Roughen both ends of the quartz rod with sandpaper. Use 2-ton epoxy or insta-cure cyanoacrylate to attach a PPMS ACMS straw adapter (part # 4084-234) to both ends of the rod. Screw a male cap (part # 4084-233) into the lower end of the straw adapter. The extra attachments on the end of the rod help minimize horizontal motion of the sample during data collection and improve experimental reproducibility.

Notes about the quartz rod

- A sample will fail if the quartz rod breaks. In normal operation, these samples have been found to be fairly robust.
- Avoid low temperatures ($T < 100$ K). The first production run of these samples used epoxy, which appeared to cause stress at the interface between the quartz and the Delron straw adapters. This stress causes the ends to break with the quartz rod still glued to the inside of the Delron straw adapters. Since the analysis occurs best at room temperature, this condition should not interfere with routine examinations.

Performing the Measurement

The measurements can be performed with either the standard DC or the preferred RSO techniques.

Using a damp cloth, wipe off fingerprints and other possible magnetic contaminants from the quartz rod, then wipe the quartz rod completely with a Kimwipe or other low lint material.

Insert the sample into an MPMS at $T=300$ K, and an applied field, $H=1.0$ T.

Initialize the transport, set the scan length to 6 cm, and run a centering scan. Use caution when running a full DC scan since (see Figure 1) the end of the quartz rod produces a signal much greater than the sample response.

Compare the value of the moment obtained in this scan with the value printed on the sample storage box label to verify the identity of the sample.

After centering the sample, change the scan parameters to those used in the measurement and optimize the center. Recommended scan parameters for RSO are: a center scan at 3 cm and 1.5 Hz with 10 cycles, auto-range and linear regression. Recommended scan parameters for DC are: a 4 cm scan with 32 data points and linear regression.

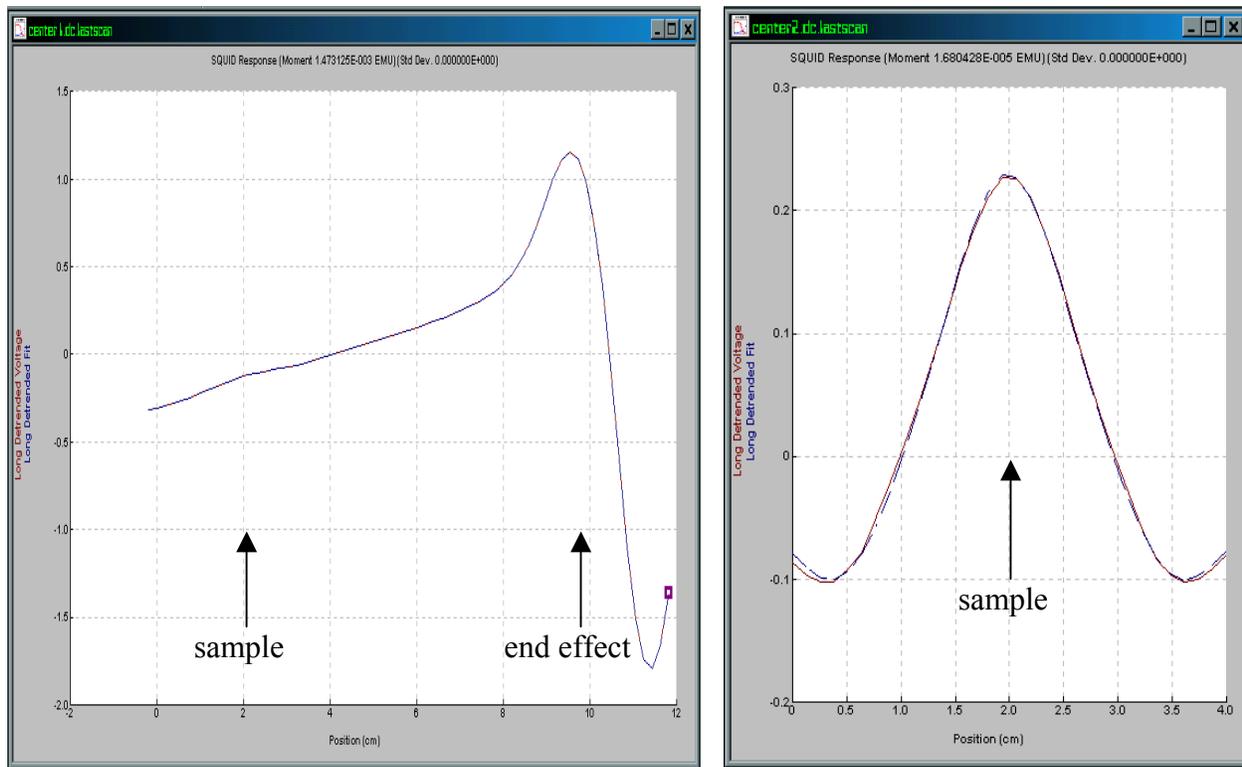


Figure 1. SQUID voltage versus scan distance for full (left) and centering (right) DC scans showing the signal generated by the end of the quartz rod is large at $H=1.0$ T and $T=300$ K in comparison to the signal from the sample.

For an immediate demonstration of the absolute sensitivity

1. Set the field to zero in high resolution oscillate mode.
2. Reset the magnet to hasten the stability of the superconducting magnet.
3. Apply a small field in the range $H=10$ to 100 Oe, which will produce a moment in the 10^{-8} emu range. The value of the field required to obtain a given value of the moment depends on the sample size. For example, if the moment at $H=1.0$ T and $T=300$ K is in the 10^{-6} emu range, then a field of $H=100$ Oe is needed to produce a moment in the 10^{-8} emu range.
4. After applying the field, wait at least five minutes before performing the measurement.

Note: In this highly sensitive region, instrumental artifacts like flux jumps and SQUID drift can cause difficulties. Generally, increasing the waiting time before a measurement is taken will stabilize the magnet and the SQUID electronics.

Improving the reliability of the data requires statistical averaging. Quantum Design recommends running a sequence that makes use of the multi-measure command. This command is available in MultiVu version 1.52 and newer software releases. Typically, the multi-measure command parameter is set to the default value of 10 measurements, which rejects and recollects up to two data points of more than two standard deviations from the average. Performing a magnetization versus field sequence overnight will also help to minimize external noise sources.

Representative Data

Since the sample is paramagnetic, a linear magnetization versus field response is expected at room temperature. Figure 2 presents the average magnetic moment and standard error values as a function of field on a log-log scale for a $12 \mu\text{g}$ sample of Er_2O_3 embedded in a quartz rod. All of the data was collected in a continuous sequence that used the RSO and multi-measure command parameters described above. After first quenching the magnet at $H=0$ Oe, the field was incrementally increased to $H=10\text{kOe}$.

Open symbols represent data taken in positive fields; solid and crossed symbols represent negative fields. A linear least square fit is shown as a solid line. The deviation from linearity at low fields is most likely due to trapped flux in the MPMS superconducting magnet. The linear behavior of the sample at low fields was confirmed on another sample having more Er_2O_3 mass by using the AC susceptibility technique. The circles represent the standard errors calculated in the multi-measure command. These values signify the relative sensitivity of the instrument.

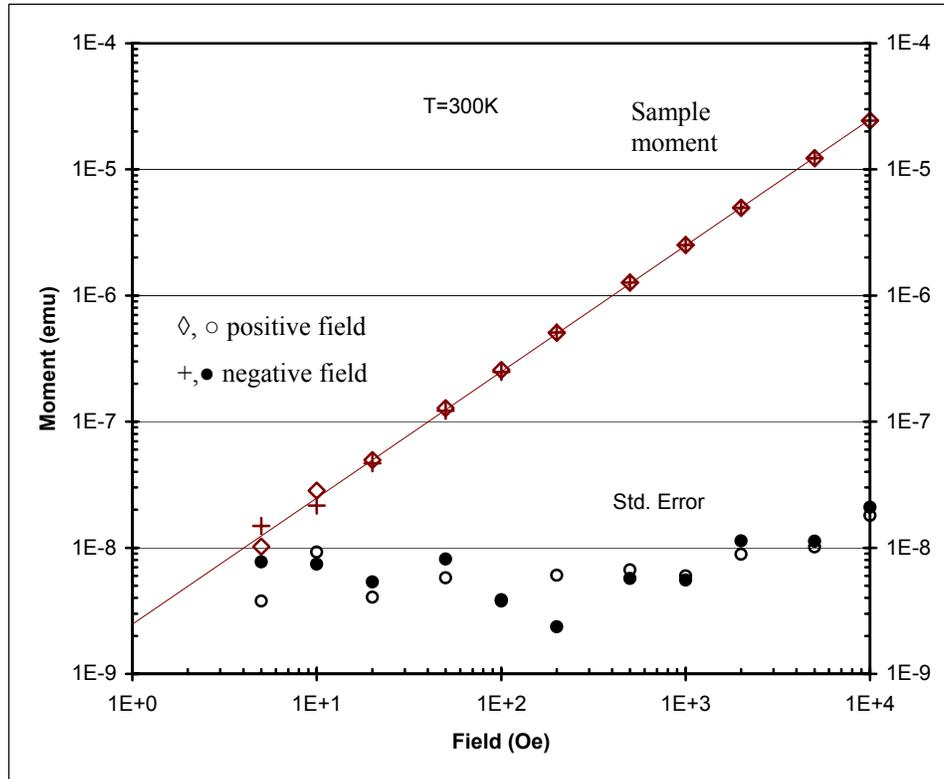


Figure 2. Average magnetic moment and standard error values for Er₂O₃ sample in a quartz rod. The open symbols are for positive fields and cross or closed symbols are for negative fields.

Data Fitting Algorithm

Use the linear regression mode when calculating the moment from the raw voltage versus position data to increase the absolute sensitivity. The iterative mode leaves the sample center position as a free variable to account for sample position changes as temperature is changed. However, when noise becomes significant, the iterative regression algorithm can mistake a noise peak as the sample center and thus gives poor reproducibility. Forcing the calculation to presume the sample is centered will improve the noise rejection and produce a more consistent analysis. For a more detailed discussion, please see MPMS Application Note 1014-203, Outline of Data Regression Routines.

Conclusion

The Er₂O₃ sample described in this application note demonstrates the capabilities of the MPMS and should not be construed as a standard. This application note has emphasized the criteria that are important when verifying the absolute sensitivity of the MPMS. The primary concern is a sample mounting technique that produces essentially no background contribution. The RSO parameters include a maximum slope option that could extend the absolute limits even further, but will sacrifice some accuracy.