



MPMS Application Note 1014-213

Subtracting the Sample Holder Background from Dilute Samples

Introduction

The MPMS MultiVu software contains an automatic background subtraction feature. This application note provides instructions for MPMS users with earlier software releases, through manual modification of the raw data. In addition, this document discusses the general fitting algorithm used to convert the raw SQUID voltage to the sample moment.

One technique for measuring the magnetization of a sample with the MPMS is to measure a sample inside a sample holder. The raw data is fit by Equation 1. The amplitude, corrected by the appropriate calibration factors, gives the magnetization of the sample plus holder. The measurement procedure is then repeated with the empty (diamagnetic) sample holder. The two magnetizations are subtracted from each other, yielding the magnetization of the sample alone.

For magnetically dilute samples, for example, biological material and model compounds, this procedure fails because the response curve of the sample often does not have the ideal pattern. This is because the paramagnetic response of the sample and the diamagnetic response of the sample holder are of the same magnitude, nearly canceling each other. Also, there is a small spatial offset between the two response curves along the axis of the magnet. This offset depends significantly on the sample volume used. Since diamagnetism is temperature independent, this problem can also occur for a paramagnetic sample even when the response looks good at one temperature.

Fitting Raw Data

The MPMS moves the sample through a set of superconducting pick up loops arranged in a second order gradiometer geometry. The only data collected is the sample position and the raw SQUID voltage. The idealized shape of the response curve is shown in Figure 1 for a sample with 0.03 emu. For a DC measurement using either the standard DC transport or the RSO transport, the best fit to this curve is found using Equation 1.

Equation 1

$$f(Z) = X(1) + X(2) \cdot Z + X(3) \cdot \{2[R^2 + (Z + X(4))^2]^{-3/2} - [R^2 + (A + (Z + X(4)))^2]^{-3/2} - [R^2 + (-A + (Z + X(4)))^2]^{-3/2}\}$$

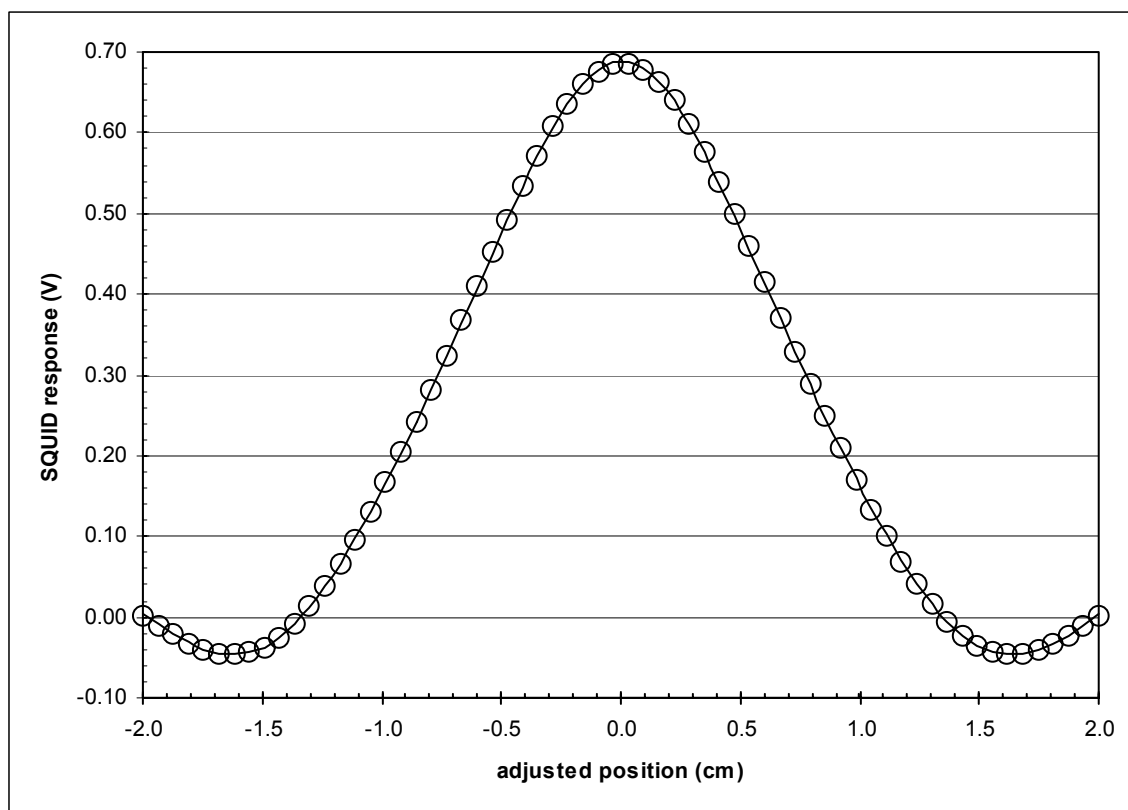


Figure 1. The circles represent the raw SQUID voltage produced during standard DC scan. The line is the fit to the response function using Equation 1.

Equation 1 represents the raw SQUID voltage ($f(z)$) as a function of the sample position (Z). The constants in this equation are the longitudinal radius ($R=0.97$ cm) and the longitudinal coil separation ($\Lambda =1.519$ cm). The fit parameter $X(1)$ is a constant offset voltage. The parameter $X(2)$ takes into account a linear electronic SQUID drift during data collection. The parameter $X(4)$ is the shift of the sample along the axis of the magnet. The magnetic moment can be calculated from the amplitude $X(3)$ by applying Equation 2.

Equation 2

$$\text{Moment (emu)} = \frac{X(3) * \text{longitudinal regression factor}}{(\text{SQUID cal. Factor} * \text{sensitivity factor} * \text{correction factor})}$$

The longitudinal regression factor and SQUID calibration factor are device dependent values. In the MultiVu software these can be found in the utilities/calibration window. The sensitivity factor is determined by the range and gain settings during the scan and is shown in the last column of Table 1. Finally, the correction factor is a constant 0.9125 and was incorporated into the software when the Equation 1 became the standard way to calculate the moment.

The fit parameters used in Figure 1 are provided in Appendix 1. Appendix 1 is a modified Microsoft Excel™ spreadsheet that shows a manual fitting operation. The fit equation is for illustration purposes only. If interested, please contact Quantum Design and the file will be sent to you.

To fit RSO data, additional concerns must be addressed. First, in the moment calculation, you must multiply Equation 2 by the RSO regression factor. This additional correction factor is found in MultiVu under the utilities/calibration/RSO window and typically has a value near unity. The second concern is the SQUID drift X(2) factor in the fitting equation. The SQUID drift is more properly considered in units of volts per time. The conversion to volts per cm in Equation 1 is appropriate for DC measurements. However, since RSO measurements start and finish in the center of the scan, this no longer is valid. A good first approximation is to use a data point array of 64 points and define the drift in units of volts per point. A sample of these modifications is provided in Appendix 2.

Data Correction

When the sample signal is comparable to the signal produced by the sample holder, the response function will not have the ideal shape. The background subtraction procedure allows the removal of the contribution from the sample holder thus producing a sample response, which can be fit using Equation 1.

1. The sample and empty sample holder must be measured under identical conditions. Care must be taken to mount the sample holder in the exact same location for both analyses. Be sure to collect raw data for all measurements.
2. Measure the empty sample holder.
3. Measure the sample + holder.
4. Convert both sets of raw SQUID voltages to scaled voltages using Equation 3.
3. Now that the data are on the same relative range, simply subtract the data sets point by point to get the sample position versus scaled data array. Then apply the fit of Equation 1 and determine the magnitude of X(3). Since the data has been scaled already, simply divide the value of X(3) by the correction factor (0.9125) and this is the sample moment in units of emu.

Equation 3

$$\text{Scaled voltage} = \frac{\text{raw voltage} * \text{longitudinal regression factor}}{(\text{SQUID cal. Factor} * \text{sensitivity factor})}$$

An illustration of this procedure is provided in Figure 2 and incorporates the data from the automatic background subtraction software in MultiVu. All MultiVu raw data files have the raw SQUID voltage and the device independent scaled voltages. In Figure 2 it can be seen that the diamagnetic sample holder has a response on the same order of magnitude as the paramagnetic sample. This produces a response function that cannot be fit. After point-by-point subtraction of the previously measured sample holder, a typical sample response function is generated which can be properly fit. The circles in Figure 2 represent the detrended scaled sample voltage which has basically incorporated the variables X(1) and X(2) from Equation 1.

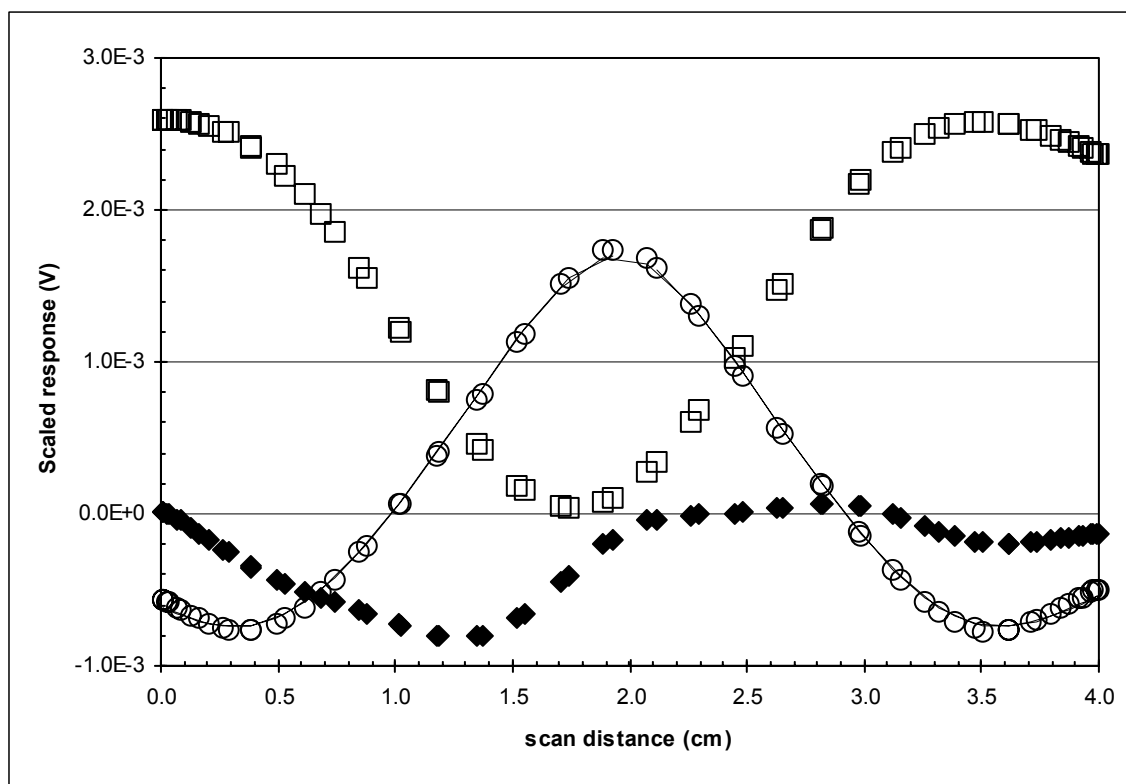


Figure 2. Data representative of a standard automatic background subtraction raw RSO data file with all voltages scaled as in Equation 2. The diamonds represent the response of a sample in a sample holder. The squares are the previously recorded scaled response for the sample holder. The circles represent the long detrended sample data after point-by-point subtraction. The line is the computer generated long detrended fit, which produces the sample moment value.

Conclusion

The MPMS is an extremely sensitive instrument. However, contributions from multiple sources can cause limitations in achieving the full potential of this sensitivity. Special sample mounting techniques can be devised to minimize the contributions from sample holders. The procedure described in this report will allow the user to further reduce the unwanted effects of the sample holder on the measurement of low moment samples.

Sensitivity (emu)	Range Code	Range	Gain Code	Gain	Sensitivity Calibration Factor [Volts/ $\mu\Phi$]
.000125	0	1x	3	10x	10.00x
.000250	0	1x	2	5x	5.00x
.000625	0	1x	1	2x	2.00x
.001250	0	1x	0	1x	1.00x
.002500	1	10x	2	5x	0.50x
.006250	1	10x	1	2x	0.20x
.012500	1	10x	0	1x	0.10x
.025000	2	100x	2	5x	0.05x
.062500	2	100x	1	2x	0.2x
.125000	2	100x	0	1x	0.01x
.250000	3	1000x	2	5x	0.005x
.625000	3	1000x	1	2x	0.002x
1.25000	3	1000x	0	1x	0.001x

Table 1 Parameters of the MPMS-SQUID from Quantum Design. Range and Gain code are numbers given in the MPMS data set of the different Range and Gain values.

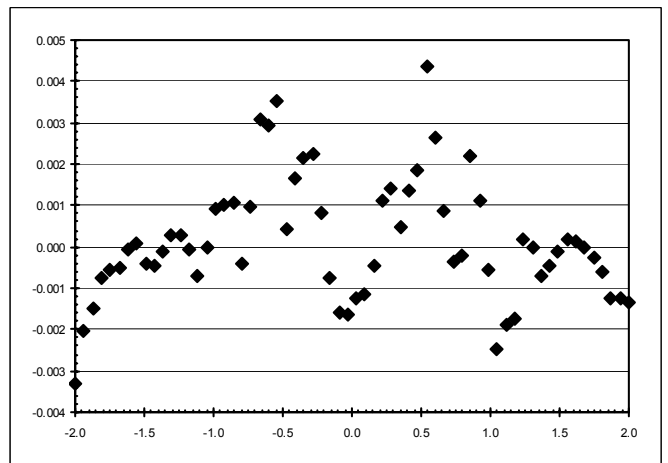
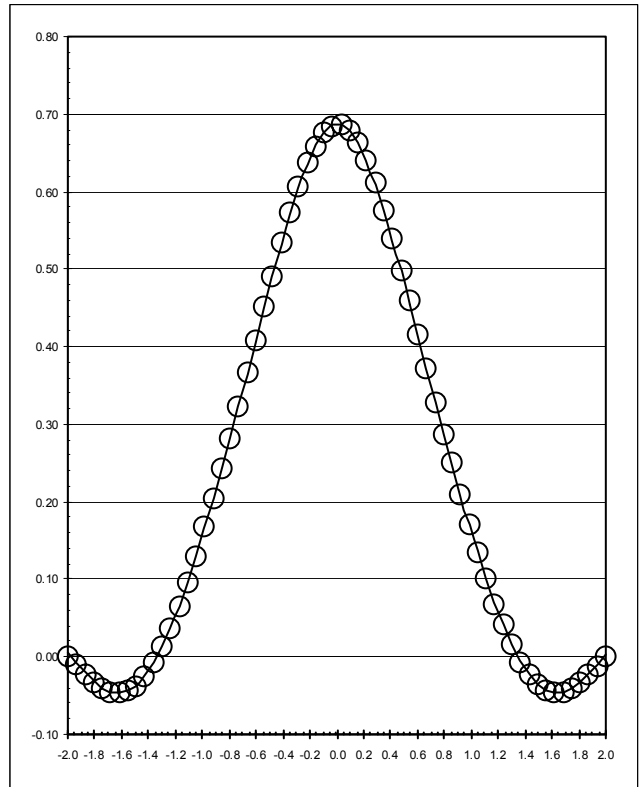
Appendix 1. Microsoft Excel™ spreadsheet showing a fit function for a standard DC raw data set.

3.22E-02 emu = target x(1) = 0.177 V lambda = 1.519 cm range = 3
 x(2) = 0.000 V/cm radius = 0.97 cm gain = 1
 x(3) = 0.276 V corr. Fact. = 0.9125 Sens. Cal = 0.002
 x(4) = -0.005 cm SQUID C = 8588

3.22E-02 emu = x(3)*long reg. Factor/(SQUID cal factor*Sensitivity Cal. Factor*Correction Factor) Long. Re. = 1.825
 RSO reg. = 1.01

$$\text{Fit} = \text{\$H\$1} + (\text{\$H\$2} * \text{B8}) + (\text{\$H\$3} * (2 * (((\text{\$L\$2}^2) + ((\text{B8} + \text{\$H\$4})^2))^{\text{-1.5}}) - (((\text{\$L\$2}^2) + ((\text{\$L\$1} + \text{B8} + \text{\$H\$4})^2))^{\text{-1.5}}) - (((\text{\$L\$2}^2) + ((-1 * \text{\$L\$1}) + \text{B8} + \text{\$H\$4})^2))^{\text{-1.5}}))$$

Raw Position (cm)	Adjusted Position (cm)	Long Voltage	Fit (V)	difference
0.00	-2.00	0.002	0.005	-0.003
0.06	-1.94	-0.010	-0.008	-0.002
0.13	-1.87	-0.022	-0.020	-0.002
0.19	-1.81	-0.032	-0.031	-0.001
0.25	-1.75	-0.040	-0.040	-0.001
0.32	-1.68	-0.045	-0.045	-0.001
0.38	-1.62	-0.047	-0.047	0.000
0.44	-1.56	-0.044	-0.044	0.000
0.51	-1.49	-0.037	-0.037	0.000
0.57	-1.43	-0.025	-0.025	0.000
0.63	-1.37	-0.008	-0.008	0.000
0.70	-1.30	0.013	0.013	0.000
0.76	-1.24	0.038	0.038	0.000
0.83	-1.17	0.066	0.066	0.000
0.89	-1.11	0.096	0.097	-0.001
0.95	-1.05	0.130	0.131	0.000
1.02	-0.98	0.167	0.166	0.001
1.08	-0.92	0.204	0.203	0.001
1.14	-0.86	0.243	0.242	0.001
1.21	-0.79	0.281	0.282	0.000
1.27	-0.73	0.323	0.322	0.001
1.33	-0.67	0.367	0.364	0.003
1.40	-0.60	0.409	0.406	0.003
1.46	-0.54	0.452	0.449	0.004
1.52	-0.48	0.491	0.491	0.000
1.59	-0.41	0.533	0.532	0.002
1.65	-0.35	0.572	0.570	0.002
1.71	-0.29	0.607	0.605	0.002
1.78	-0.22	0.636	0.636	0.001
1.84	-0.16	0.659	0.660	-0.001
1.90	-0.10	0.676	0.677	-0.002
1.97	-0.03	0.685	0.687	-0.002
2.03	0.03	0.686	0.687	-0.001
2.10	0.10	0.678	0.679	-0.001
2.16	0.16	0.663	0.663	0.000
2.22	0.22	0.641	0.640	0.001
2.29	0.29	0.612	0.610	0.001
2.35	0.35	0.576	0.576	0.000
2.41	0.41	0.539	0.538	0.001
2.48	0.48	0.499	0.497	0.002



Appendix 2. Microsoft Excel™ spreadsheet showing a fit function for a standard RSO raw data set.

3.22E-02 emu = target x(1) = -0.502 V lambda = 1.5190 cm range = 3
 x(2) = 0.000 V/pt radius = 0.97 cm gain = 1
 x(3) = 0.273 V corr. Fact. = 0.9125 Sens. Cal = 0.002
 x(4) = -0.005 cm SQUID C = 8589

3.22E-02 emu = x(3)*long reg. Factor*RSO reg. Factor/(SQUID Cal. Factor*Sensitivity Cal. Factor*Fit fudge Factor)
 Long. Re. = 1.825
 RSO reg. = 1.011

$$\text{Fit} = \text{\$H\$1} + (\text{\$H\$2} * \text{D8}) + (\text{\$H\$3} * (2 * (((\text{\$L\$2}^2) + ((\text{B8} + \text{\$H\$4})^2))^{-1.5}) - (((\text{\$L\$2}^2) + ((\text{\$L\$1} + \text{B8} + \text{\$H\$4})^2))^{-1.5}) - (((\text{\$L\$2}^2) + ((-1 * \text{\$L\$1}) + \text{B8} + \text{\$H\$4})^2))^{-1.5})))$$

Raw Position (cm)	Adjusted Position (cm)	Long Voltage	Fit (V)	difference	
1.90	-0.10	-0.012	1	-0.008	-0.004
1.72	-0.28	-0.074	2	-0.078	0.004
1.53	-0.47	-0.179	3	-0.188	0.009
1.35	-0.65	-0.299	4	-0.309	0.010
1.17	-0.83	-0.417	5	-0.424	0.008
0.99	-1.01	-0.523	6	-0.528	0.005
0.82	-1.18	-0.610	7	-0.615	0.005
0.66	-1.34	-0.675	8	-0.678	0.003
0.51	-1.49	-0.713	9	-0.714	0.000
0.37	-1.63	-0.726	10	-0.723	-0.003
0.25	-1.75	-0.721	11	-0.716	-0.005
0.15	-1.85	-0.708	12	-0.702	-0.006
0.08	-1.92	-0.694	13	-0.688	-0.006
0.03	-1.97	-0.684	14	-0.678	-0.006
0.00	-2.00	-0.679	15	-0.673	-0.007
0.00	-2.00	-0.680	16	-0.673	-0.007
0.03	-1.97	-0.684	17	-0.678	-0.006
0.07	-1.93	-0.694	18	-0.687	-0.007
0.13	-1.87	-0.705	19	-0.698	-0.006
0.21	-1.79	-0.715	20	-0.711	-0.005
0.30	-1.70	-0.724	21	-0.720	-0.004
0.40	-1.60	-0.727	22	-0.723	-0.004
0.51	-1.49	-0.718	23	-0.714	-0.005
0.62	-1.38	-0.693	24	-0.688	-0.005
0.75	-1.25	-0.648	25	-0.643	-0.004
0.89	-1.11	-0.582	26	-0.580	-0.003
1.04	-0.96	-0.501	27	-0.499	-0.002
1.20	-0.80	-0.404	28	-0.404	-0.001
1.37	-0.63	-0.295	29	-0.295	0.000
1.54	-0.46	-0.181	30	-0.180	-0.001
1.72	-0.28	-0.079	31	-0.074	-0.005
1.91	-0.09	-0.015	32	-0.007	-0.008
2.10	0.10	-0.011	33	-0.006	-0.006
2.28	0.28	-0.071	34	-0.072	0.001

