Quantum Design

# Magnetic Property Measurement System

## SQUID VSM AC Option User's Manual

**Part Number 1505-400, A1**

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## Trademarks

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P R E F A C E

# Contents and Conventions

## P.1 Overview

In this chapter we describe the scope of the manual, the conventions used and most importantly the safety guidelines. Since the SQUID VSM uses cryogens and high power components, we strongly recommend to be aware of all hazards in order to prevent injuries and system damage.

## P.2 Scope of the Manual

This manual explains how to use the SVSM AC option software and hardware, how to perform AC sample measurements, and perform basic troubleshooting.

## P.3 Conventions in the Manual

| | |
|---|---|
| **File menu** | Bold text identifies the names of menus, dialogs, options, buttons, and panels used in the SQUID VSM MultiVu software. |
| **File > Open** | The > symbol indicates that you select multiple, nested software options. |
| `.dat` | The Courier font indicates file and directory names and computer code. |
| **Important** | Text is set off in this manner to signal essential information that is directly related to the completion of a task. |
| **Note** | Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances. |

This symbol signals specific caution or conditions that could result in system damage, bodily harm, or loss of life.

This symbols signals electrical hazards that could result in bodily harm, or loss of life. Used at all accessible 200-230 V power outlets.

This symbol signals **cryogenic hazards** that could result in bodily harm and loss of life. Used wherever accessible parts could reach temperatures below 0°C (32°F).

This symbol signals **hot surface hazards** that could result in bodily harm and loss of life. Used wherever accessible parts could reach temperatures above 60°C (140°F).

This symbol signals information on **fusing**.

# P.4 Safety Guidelines and Regulatory Information

Before using this product, please read the entire content of this User’s Manual and observe all instructions, warnings and cautions. These are provided to help you understand how to safely and properly use the SQUID VSM and reach its best performances.

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse or improper operation of the system. Please contact your Quantum Design representative for any service issues.

This product is NOT operator-serviceable.

Observe the following safety guidelines when you use your system:

- In case of emergency, switch the power off at the rear of the main cabinet as well as the helium compressor or unplug the main power cords from the laboratory power outlet.
- To prevent electrical shock, unplug the system before you install it, adjust it, or service it.
- The compressor must be wired into a dedicated circuit breaker (see supplied compressor manual for electrical requirements).
- For continued protection against fire hazard, electric shock and irreversible system damage, replace fuses only with same type and rating of fuses for selected line voltage. Information about user-accessible fuses and their replacement is summarized in Appendix A.3.9 of your SVSM User's manual.
- Direct contact with cryogenic liquids, materials recently removed from cryogenic liquids, or exposure to the boil-off gas can freeze skin or eyes almost instantly, causing serious injuries similar to frostbite or burns. Wear protective gear, including clothing, insulated gloves, and eye protection, when you handle cryogenic liquids.
- Transfer cryogenic liquids only in areas that have adequate ventilation and a supply of fresh air. Nitrogen and helium gas can displace the oxygen in a confined space or room, resulting in asphyxiation, dizziness, unconsciousness, or death.
- Keep this system away from radiators and heat sources. Provide adequate ventilation to allow for cooling around the cabinet and pump console. The distance between the system and wall should be at least 30 cm. (12 inches) in each direction. Do not obstruct the ventilation openings on the top of the cabinet.
- Do not obstruct the ventilation outlet located on the left side of the pump console and air intake at the rear. The clearance around the pump console should be at least 20 cm. (8 inches) in each direction.
- Do not obstruct or pinch the pump exhaust line located at the rear of the pump console.

C H A P T E R 1

# Introduction to the AC Measurement Option

## 1.1 Introduction

- Section 1.2 presents an overview of the operation of the AC measurement option.
- Section 1.3 explains the theory of operation of the AC option
- Section 1.4 discusses the AC option hardware.

## 1.2 Overview of the AC Option

In an AC susceptometer, an oscillating AC magnetic field is applied to the sample. The change in flux seen by the detection circuitry is caused only by the changing magnetic moment of the sample as it responds to the applied AC field. The differential or AC susceptibility of $\chi_{ac} = \partial M/\partial H$ obtained from these measurements is described as having both real and imaginary components $\chi'$ and $\chi''$, where the imaginary component is proportional to the energy losses in the sample. The complex susceptibility can provide various types of information on properties such as the structural details of materials, resonance phenomena, electrical conductivity by induced currents, relaxation processes such as flux profiles and flux creep in superconductors, and energy exchange between magnetic spins and the lattice in paramagnetic materials.

### 1.2.1 Advantages of the SVSM AC System compared to Conventional AC Susceptometers

Conventional AC susceptometers measure the voltage induced in an inductive detection coil by an oscillating AC magnetic moment. The most common systems use mutual inductance bridges to measure the voltages induced, and some use digital processing to improve noise rejection. These systems measure only signals with frequencies at or very near the applied excitation, so sensitivity is greatly increased by reducing the effective noise level to that in the measurement bandwidth centered on the frequency of interest. However, conventional systems lose sensitivity at low frequencies because the voltage induced in the detection coil is proportional to the frequency of the oscillating drive field.

The SVSM AC option solves this problem by combining an AC drive field with a SQUID-based detection system. The SQUID (Superconducting QUantum Interference Device) is an extremely sensitive flux-to-voltage converter that directly measures the change in flux caused by the change in magnetization of the sample inside a superconducting detection coil coupled to the SQUID circuit. The frequency-independent coupling between magnetic flux and induced currents in the superconductors allows use of AC frequencies and AC drive fields that are many orders of magnitude lower than those used in conventional AC systems.

In the SVSM AC system, a set of additional drive coils is used to generate the AC field in the sample chamber independently of the field in the main (superconducting DC) magnet. The drive coils are situated in the helium bath between the main magnet and the gradiometer detection coils, concentric with the superconducting magnet, with all of these components combined into a rigid structure to minimize changes in coil geometry as the sample temperature and DC field are varied.

The AC Option requires additional hardware (the SVSM AC CAN module model CM-K) and an extensive system calibration to remove effects based on the intrinsic properties of the probe. All operations necessary for taking AC measurements are integrated into the SVSM MultiVu software application. The SVSM AC option automatically controls operations during a measurement; user input is required only to initiate the measurement.

# 1.3 Theory of Operation

## 1.3.1 AC Magnetometry

The standard measurement employed by the SVSM measures the DC magnetic moment of a sample. DC magnetic measurements determine the instantaneous value of the magnetic moment in a sample. The sample is magnetized by a constant magnetic field and the magnetic moment of the sample is measured, producing a DC magnetization curve *M(H)*. The moment is measured by force, torque or induction techniques, the last being the most common in modern instruments like the SVSM. Inductive measurements are performed by moving the sample relative to a set of pickup coils, either by vibration or one-shot extraction. In conventional inductive magnetometers, one measures the voltage induced by the moving magnetic moment of the sample in a set of copper pickup coils. A much more sensitive technique uses a set of superconducting pickup coils and a SQUID to measure the current induced in superconducting pickup coils, yielding high sensitivity that is independent of sample speed during extraction. Inductive magnetometers can also be used to perform AC magnetic measurements.

In AC magnetic measurements, a small AC drive magnetic field is superimposed on the DC field, causing a time-dependent moment in the sample. The field of the time-dependent moment induces a current in the pickup coils, allowing measurement without sample motion. The detection circuitry is configured to detect only in a narrow frequency band, normally at the fundamental frequency (that of the AC drive field).

In order to understand what is measured in AC magnetometry, first consider very low frequencies (e.g., 1 Hz), where the measurement is most similar to DC magnetometry. In this case, the magnetic moment of the sample follows the *M(H)* curve that would be measured in a DC experiment. As long as the AC field is small, the induced AC moment is $M_{AC} = (\partial M/\partial H) \cdot H_{AC} \sin(\omega t)$ where $H_{AC}$ is the amplitude of the driving field, ω is the driving frequency, and $\chi = \partial M/\partial H$ is the slope of the *M(H)* curve, called the susceptibility. The susceptibility is the quantity of interest in AC magnetometry.

As the applied DC magnetic field is changed, different parts of the *M(H)* curve are accessed, giving a different susceptibility. One advantage of the AC measurement is already evident: the measurement is very sensitive to small changes in *M(H)*. Since the AC measurement is sensitive to the slope of *M(H)* and not to the absolute value, small magnetic shifts can be detected even when the absolute moment is large.

At higher frequencies, the AC moment of the sample does not follow along the DC magnetization curve due to dynamic effects in the sample. For this reason, the AC susceptibility is often known as the dynamic susceptibility. In this higher frequency case, the magnetization of the sample may lag behind the drive field, an effect that is detected by the magnetometer circuitry. Thus, the AC magnetic susceptibility measurement yields two quantities: the magnitude of the susceptibility, χ, and the phase shift, φ (relative to the drive signal). Alternately, one can think of the susceptibility as having an in-phase, or real, component $\chi'$ and an out-of-phase, or imaginary, component $\chi''$. The two representations are related by

$$\chi' = \chi \cos \varphi, \qquad \chi'' = \chi \sin \varphi \tag{1}$$

$$\chi = (\chi'^2 + \chi''^2)^{1/2}, \qquad \varphi = \mathrm{atan2}(\chi'', \chi') \tag{2}$$

where atan2() is the two-argument arctan() function.[1]

In the limit of low frequency where AC measurement is most similar to a DC measurement, the real component χ' is just the slope of the M(H) curve discussed above. The imaginary component, χ" , indicates dissipative processes in the sample. In conductive samples, the dissipation is due to eddy currents. Relaxation and irreversibility in spin-glasses give rise to a nonzero χ" . In ferromagnets, a nonzero imaginary susceptibility can indicate irreversible domain wall movement or absorption due to a permanent moment. Also, both χ' and χ" are very sensitive to thermodynamic phase changes, and are often used to measure transition temperatures. AC magnetometry allows one to probe all of these interesting phenomena. Typical measurements to access this information are χ vs. temperature, χ vs. driving frequency, χ vs. DC field bias, and χ vs. AC field amplitude.

## 1.3.2 SVSM AC Measurement Details

The method employed by the SVSM to measure the AC susceptibility of a sample is a multi-step process. As a first step, the software nulls the signal generated by the AC field coupling, field non-uniformity, and gradiometer imbalance (this step is only done once for a given amplitude and frequency setting). The next steps comprise of acquiring the AC signal generated by the sample itself in different positions with respect to the gradiometer in order to extract the sample's AC susceptibility.

For the first step of the measurement (referred to as "nulling") the sample is positioned in between the bottom two coils of the gradiometer, in the location where the signal detected by the gradiometer is zero (see Figure 4-1 in the main SVSM User's Manual). The software then applies an AC field of the requested frequency and amplitude to the sample using the AC or modulation coil. The AC response from the SQUID is monitored and a nulling waveform, which will cancel the AC response, is calculated. The nulling waveform is injected into a separate coil and the software will automatically adjust this signal to minimize the resulting AC signal seen by the

[1] atan2(y, x) = arctan(y/x) for x > 0; atan2(y, x) = arctan(y/x) + 180° for x < 0

SQUID. This nulling allows the software to make use of the full dynamic range of the SQUID to measure the sample signal and thus achieve better sensitivity for small signals.

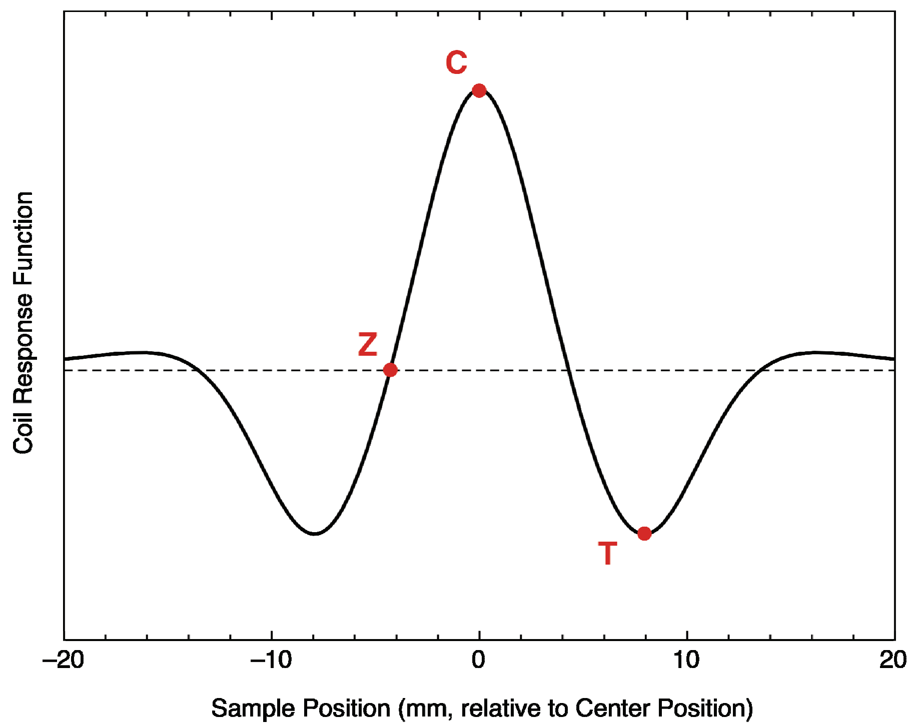


Figure 1-1 Positions used for data acquisition in AC measurements

Once the nulling operation is complete, the sample is positioned in various places within the gradiometer – the exact order and location of those points depends on the measurement type chosen by the user. The positions are based on the response function of the gradiometer and comprise of the bottom zero-crossing (Z), the center position (C), and the position near the top gradiometer winding where the response has a maximum (T) – see Figure 1-1 for a graphical view of these measurement positions. At each of these positions, the signal response from the SQUID is measured for the specified amount of time or number of wave forms and the AC response is analyzed with a lock-in technique. This yields the in-phase and out-of-phase components $M_i'$ and $M_i''$ of the magnetization at each position $i$:

$$M_i = M_i' \cos(\omega t) + M_i'' \sin(\omega t) \qquad (3)$$

Note that the actual signal recorded by the SQUID typically also has an additional DC offset as well as linear drift, both of which are rejected by the employed lock-in technique.

At each of the various positions, the measured AC moment is a combination of the sample moment $M_S$ and the background moment $M_{BG}$ (the residual signal left over after the nulling procedure). Based on the known geometry and response function of the gradiometer, the expected values at the various positions are – assuming that both the background and sample signals are constant over the time frame of the measurement:

$$\begin{aligned} M(Z) &= M_{BG} \\ M(C) &= M_S + M_{BG} \\ M(T) &= aM_S + M_{BG} \end{aligned} \qquad (4)$$

where the factor *a* is determined by the geometry of the gradiometer (and calculates to about -0.4 in the case of the SVSM). Without the initial nulling, the background signal $M_{BG}$ can be multiple orders of magnitude larger than the sample signal $M_S$.

Using multiple data points in the various locations inside the gradiometer, the software then extracts the sample moment $M_S$. Standard measurement sequences used by the software are “C-T-C” (three-point measure) and “Z-C-T-C-Z” (five-point measure). The three-point measurement corrects for drifts of the sample moment over the course of the measurement, whereas the five-point measurement corrects for drifts of both, the sample and the background signal. Each measurement with a new set of parameters (AC amplitude or frequency) automatically triggers a nulling operation (at the “Z” position) before the first signal measurement. See section Section 2.5 for a discussion on how to chose the best measurement type for a given experiment

# 1.4 System Hardware

The differences between a standard system and a system with the AC Option installed as well as each of the major components of the AC Option are described in the following sections.

## 1.4.1 Differences between a Standard System and a System with AC Option installed

The AC Option seamlessly integrates into the standard system, allowing the user to perform all measurements with the familiar user interface. While the next chapter will describe changes in the software, this chapter explains the additional hardware that is part of the AC Option.

Once the AC Option is installed on a system, it will be permanently enabled and no special user interaction has to be performed to use it. In addition, it doesn’t affect the normal (VSM) measurements, so that standard system operation is identical to that of a system without the AC Option.

## 1.4.2 AC Module

All operations of the SVMS AC Option are performed with the SVSM AC Module (Model CM-K). The module contains the current drivers to generate the AC drive fields as well as a high-speed dedicated analog to digital converter to measure the resulting SQUID signal in order to extract the in-phase and quadrature components of the signal. These calculations are done by a digital signal processor (DSP) using a lock-in technique.

In addition to the AC current drivers the module also contains a set of DC current drivers for interoperability with the Ultra-Low Field Option (if present on the system).

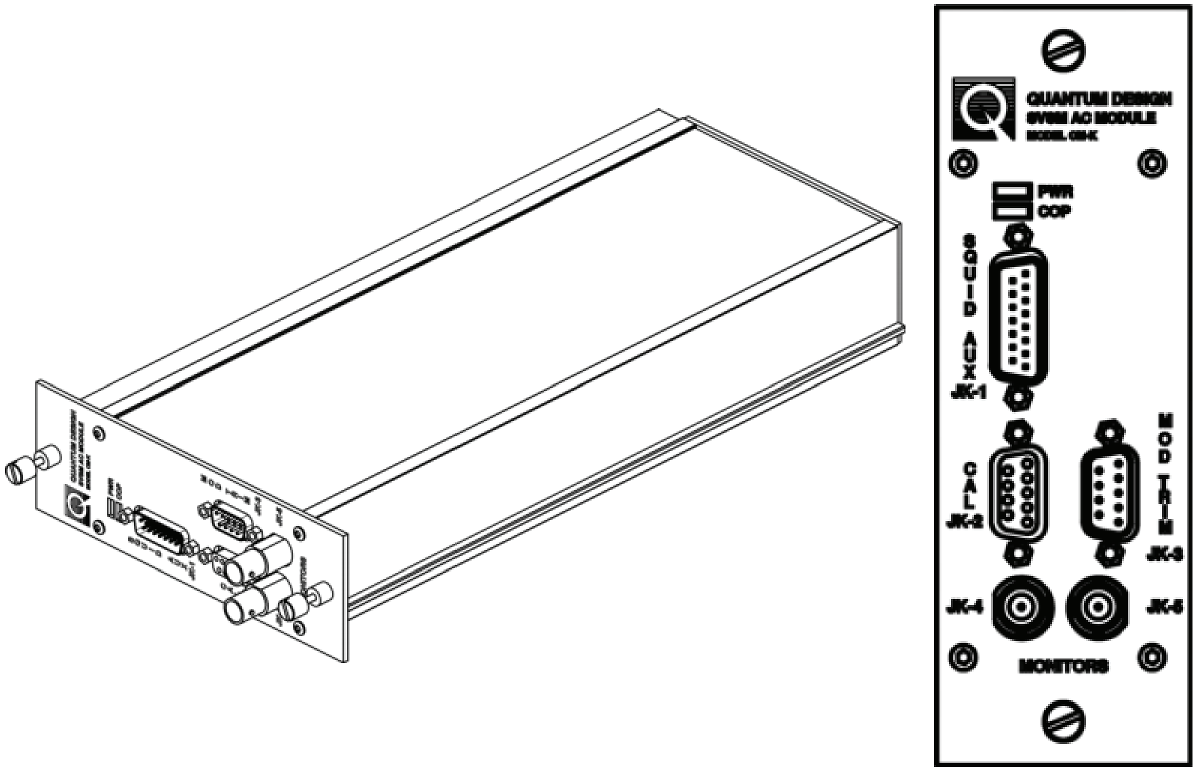


Figure 1-2 AC Module – full and front panel view

The AC Module is a high-power module (see Figure 3-4 in the main SVSM User Manual) and needs to be installed into one of the high-power (leftmost) bays in the CAN tower.

To avoid damage to the module, only install or remove CAN modules from the CAN tower if the power on the power drawer has been turned off.

## 1.4.3 System Connections

In order to perform AC measurements, the following connections have to be established (see also the "System Interconnects" section in Appendix D of the main SVSM User Manual):

Table 1-1 AC Module front panel connections and signal descriptors

| Connector (designator) | Function | Connects to (designator) | Cable used (QD Part No) |
|---|---|---|---|
| SQUID AUX (JK-1) | SQUID signal and reset control | SQUID Module (JF-4) | 3101-404 |
| CAL (JK-2) | (service use only) | N/A | |
| MOD TRIM (JK-3) | Current output to AC coils | Power Drawer (JQA-3) | 3101-402 |
| MONITOR (JK-4) [2] | AC drive signal | | |
| MONITOR (JK-5) [2] | SQUID response signal | | |

For systems which also have the Ultra-Low Field Option installed, the AC Module takes precedence over the ULF Module and JQA-3 on the power drawer has to be permanently connected to JK-3 on the AC Module rather than JJ-2 on the ULF Module – there is no need to change the cabling back and forth when using both options, the software will automatically take care of using the correct current drivers.

[2] The module can display a variety of signals on the monitor BNC connectors – the values shown in the table denote the default outputs

C H A P T E R 2

# AC Option Software

## 2.1 Introduction

- Section 2.2 describes the software changes for the AC Option compared to a standard system
- Section 2.3 details the new AC Measurement window added to MultiVu by the AC Option
- Section 2.4 explains how to use the AC Option sequence commands
- Section 2.5 talks about best practices for data acquisition and sequence writing in order to use the system in an optimal way

## 2.2 Overview

The AC Option allows for two ways to perform measurements of the AC magnetization – using an immediate measurement initiated by the user or using new options in the standard measurement sequence commands. If you have the AC Option installed, the additional software features will automatically be available from within the application.

The main additions and changes to MultiVu for a system with the AC Option are:

- New immediate AC Measurement command
- Additional options available for the "Moment vs. Temperature" and "Moment vs. Field" sequence commands
- Additional data columns available in measurement data files

## 2.3 Immediate Measurements

Immediate measurements allow one to quickly measure the current AC magnetization of a sample using a single set of parameters – this is useful to get a quick idea of the sample properties and determine the best settings to be used in a sequence.

## 2.3.1 The AC Magnetization Window

Open the AC Magnetization window by selecting "Measure > AC Measurement…" from the menu:

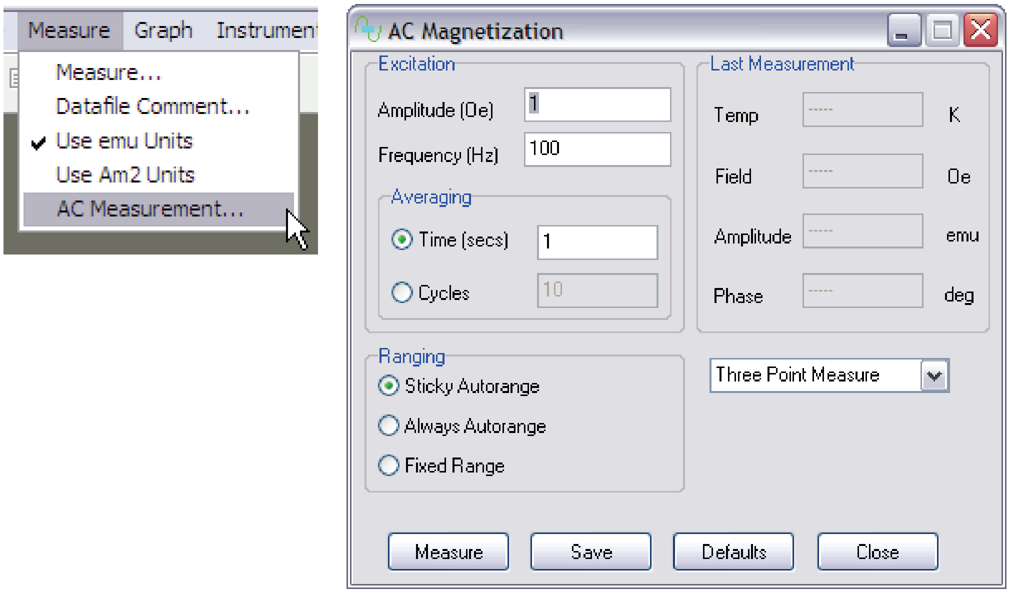


Figure 2-1 Immediate AC Measurement menu item and AC Magnetization window

The individual settings and reported values in the dialog are explained below.

The settings in the **Excitation** area of the dialog box indicate the following:

**Amplitude (Oe):** peak amplitude of the AC drive field to be applied
*Note that the actual applied field might be reduced when measuring at higher frequencies – see section 2.3.2 below*

**Frequency (Hz):** AC frequency for the drive field
*Note that the actual measurement frequency can deviate from the entered value due to limited frequency resolution in the hardware. The actual frequency will be as close as possible to the requested value*

**Averaging:** averaging time (in seconds) or number of cycles (individual waveforms) to be used for data acquisition
*Minimum averaging time is time equivalent to one cycle at the requested frequency. Actual averaging time can vary from requested value in order to accommodate an integer number of cycles. Note that the averaging time is per individual data point – e.g., a three-point measurement will require at least three times the selected averaging time*

The meaning of the various choices in the **Ranging** area of the dialog box are as follows:

**Sticky Autorange:** (preferred selection) the software tries to use the optimal range for a given moment while at the same time trying to minimize the number of range changes. This can result in a few data points being acquired in a less sensitive range than what would be possible but will typically reduce the amount of time between individual data points

**Always Autorange:** the software will make sure that every data point is measured in the most sensitive range possible. This typically adds additional time to the data collection

**Fixed Range:** all data will be collected using a fixed SQUID range as specified by the user. Note that this can result in no data being collected at all in case the signal exceeds the maximum value for the selected range.

To the right of the Ranging area the measurement type can be selected – in addition to the three-point and five-point measurements described in section 1.3, it is also possible to measure using a one-point measurement. This will collect a single data point with the sample located at the center of the gradiometer. As this measurement will not correctly remove any background signals, it is only appropriate for relative measurements where data density or keeping the sample stationary is of utmost concern.

The **Last Measurement** area on the right of the window will show the results from the last measurement (if available) – the displayed field is the current DC field applied by the main magnet.

The buttons at the bottom of the window perform the following actions:

**Measure:** acquire a single data point given the parameters selected

**Save:** save the last measurement into the current data file

**Defaults:** reset the measurement parameters to their respective default values

**Close:** close the AC Magnetization window

## 2.3.2 Dynamic Amplitude Limits

While the SVSM AC Option can typically measure with peak amplitudes up to 10 Oe, the maximum drive amplitude is limited at higher frequencies. Figure 2-2 shows the typical maximum available drive amplitude as function of frequency:

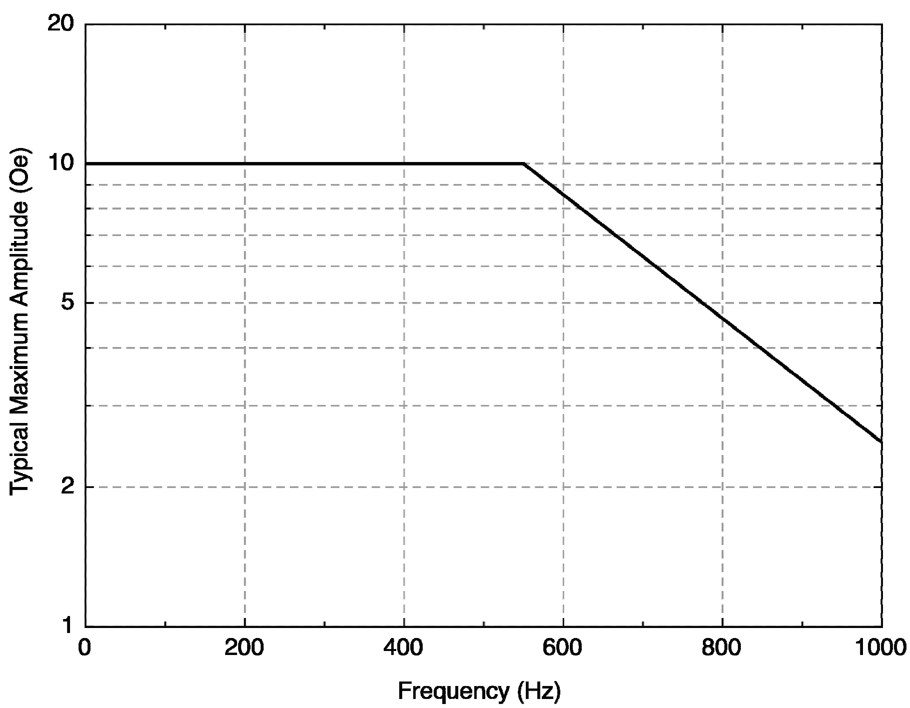


Figure 2-2 Typical maximum AC drive amplitude as function of frequency

The AC Option software automatically limits the AC drive to the maximum allowable value – when requesting a drive amplitude above the maximum value for the current frequency, the actual drive amplitude used for the measurement and recorded in the data file will be the maximum allowable value. In this case, the AC Magnetization window (Figure 2-1) will display an indicator that the drive amplitude has been reduced from the requested value.

# 2.4 Sequence Commands

For automated measurements using sequences, the AC Option includes new sequence commands and extends the functionality of some of the existing commands.

## 2.4.1 AC Magnetization

For AC measurements at stable field and temperature, a new "AC Measurement" sequence command is available (Measurement > AC > AC Magnetization). This command is similar to the immediate command described earlier in section 2.3.1 but allows for measuring using multiple parameters within a single command.

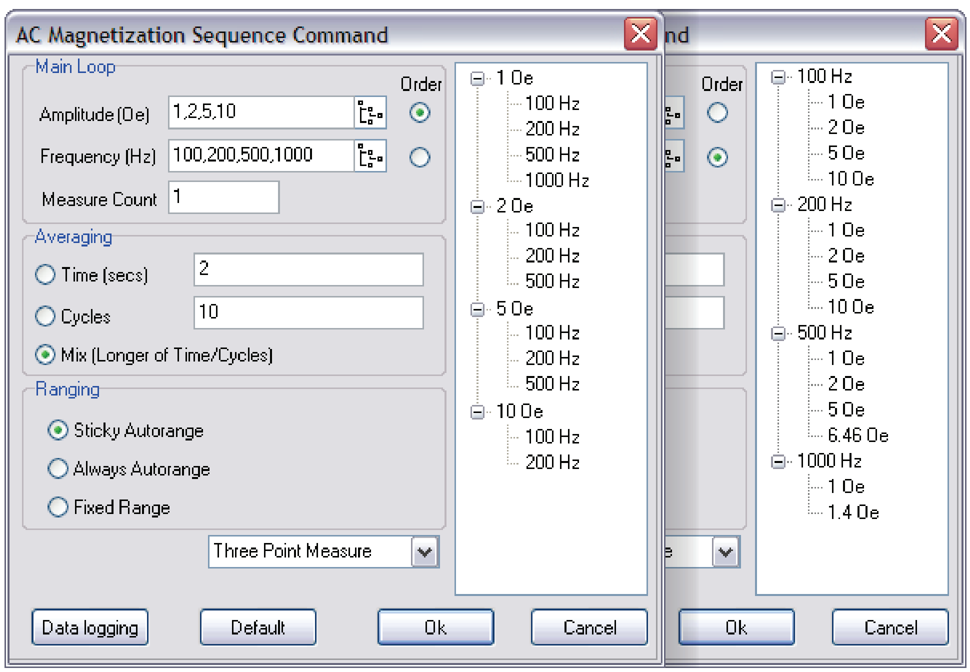


Figure 2-3 AC Magnetization sequence command (showing the resulting measurement tree for either option selected for "Order" of the measurement loop)

The **Main Loop** section of the dialog box defines the set of parameters to be used for the measurement:

**Amplitude (Oe):** list of peak amplitudes, individual values separated by commas. Clicking the tree icon at the right side of the text field allows to specify a range and number of steps to automatically populate the list of amplitudes.
*Note that the actual applied field might be reduced when measuring at higher frequencies – see section 2.3.2. The measurement tree shown on the right of the dialog will show the actual*

*amplitudes and frequencies that will be used based on the dynamic amplitude limits.*

**Frequency (Hz):** list of AC frequencies, individual values separated by commas. Clicking the tree icon at the right side of the text field allows to specify a range and number of steps to automatically populate the list of frequencies.
*Note that the actual measurement frequency can deviate from the entered value due to limited frequency resolution in the hardware. The actual frequency will be as close as possible to the requested value*

**Order:** checkbox to indicate over which parameter the measurement should loop over first – the resulting measurement sequence will be shown as a tree on the right of the dialog (the screen shot in the figure above shows the resulting measurement sequences based on the two options for "Order")

**Measure Count:** number to indicate how many times each measurement should be repeated
*Note that the whole measurement sequence as shown on the right will be repeated, not the individual measurements. This allows to investigate relaxation effects while at the same time measuring with multiple parameters*

The software will automatically perform a nulling operation for each parameter change which will require additional time for the measurement to be completed.

The meaning of the various choices in the **Averaging** area of the dialog box are as follows:

**Time (secs):** averaging time (in seconds) to be used for data acquisition

**Cycles:** number of cycles to be used for data acquisition

**Mix (Longer of Time/Cycles):** use either the specified averaging time or the specified number of cycles, whichever results in the longer time:

Example:
Selected Frequencies: 1, 2, 5, 10, 20, 50, 100
Selected Averaging Time: 1s
Selected Number of Cycles: 10
Resulting averaging times: 10s (1Hz), 5s (2Hz), 2s (5Hz), 1s (10Hz and above)

This allows acquiring data over a large range of frequencies without having to sacrifice averaging for low frequencies or measurement time for high frequencies
*Minimum averaging time is time equivalent to one cycle at the requested frequency. Actual averaging time can vary from requested value in order to accommodate an integer number of cycles. Note that the averaging time is per individual data point – e.g., a three-point measurement will require at least three times the selected averaging time*

The **Ranging** and **Measure Type** (one-point, three-point, five-point) selections are identical to the immediate AC measurement described above.

The **Data Logging** button allows to select a QMAP file for additional custom data logging (see B.3.3 in the main SQUID VSM User Manual).

## 2.4.2 Moment vs. Temperature & Moment vs. Field

When the AC Option is installed on a system, the "Moment vs. Temperature" and "Moment vs. Field" sequence commands that are part of the standard (VSM) measurement are extended to allow for AC measurements using the same simple sequence commands. The commands even allow for measuring both, AC susceptibility and DC moment, in one single temperature sweep or field loop.

With the AC Option installed, both these sequence commands will show a new tab at the top labeled "AC Settings" allowing to specify measurement parameters for AC susceptibility measurements. In addition, the "VSM Settings" tab will show a new checkbox allowing to disable VSM (DC magnetization) measurements. Apart from this additional checkbox, the

"Setup", "VSM Settings", and "Advanced" tabs in the dialog are identical to what is described in the main SQUID VSM User Manual.

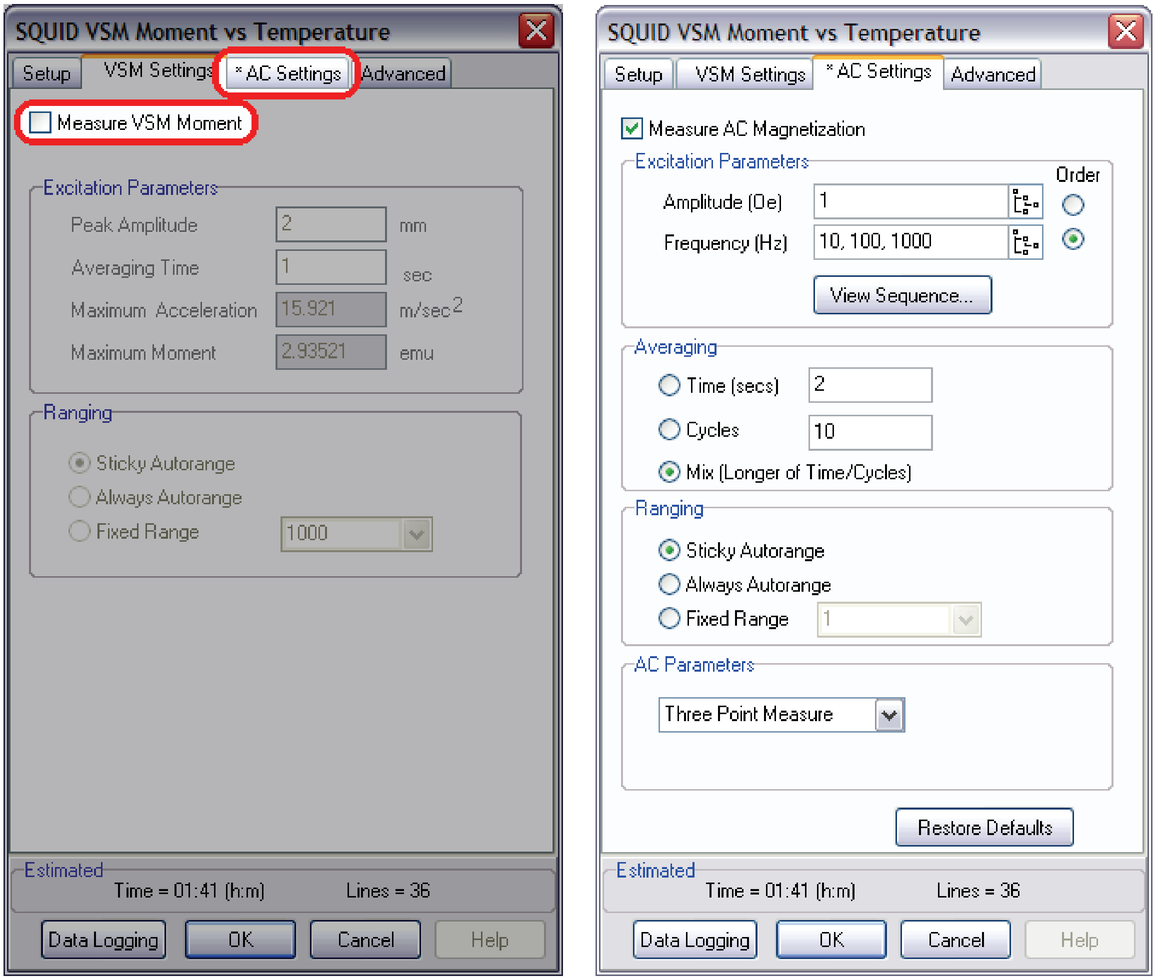


Figure 2-4 "Moment vs. Temperature" sequence command window. Highlighted on the left are the additions to the standard dialog, the right shows the new tab for AC measurement settings

The settings in the "Setup" and "Advanced" tabs as well as estimated time and number of data points at the bottom of the window are general settings that apply to either type of measurement. Both the "VSM Settings" and "AC Settings" will show a little star (as seen on the "AC Settings" and missing from the "VSM Settings" tab in the figure above) when the respective type of measurement is enabled. The "AC Settings" pane for the "Moment vs. Field" sequence command is identical to the one for the "Moment vs. Temperature" shown.

For a description of each of the settings in the "AC Settings" tab refer to the description for the "AC Magnetization" sequence command in Section 2.4.1. The "View Sequence" button shows the measurement sequence based on the selected parameters in a separate window (sequence is shown in the .same manner as inside the "AC Magnetization" sequence command). Note that averaging times and SQUID ranges can be defined separately for VSM and AC measurements.

When acquiring both VSM and AC data within a single command, the software will automatically optimize the order of operations to minimize the total time required to take the data. The software will typically alternate two VSM measurements with two AC measurement in order to minimize the additional time required to start and stop the motor vibration. When measuring while sweeping temperature, this will result in non-uniform spacing of data points for one type of measurement as function of temperature.

# 2.5 Best Practices for Data Acquisition and Sequence Writing

In order to get the most out of the system and leverage its capabilities to the fullest, a few points should be kept in mind while writing sequences and performing measurements. The following section will focus on a few points and explain how to typically achieve the best results within the shortest amount of time.

## 2.5.1 Selection of the Correct Multi-Point Measurement Type

As described in Section 1.3.2, a typical AC measurement performs a multi-step measurement to extract the sample susceptibility and discard any signal from non-perfect nulling or background (e.g., as introduced by the sample holder). Obviously this will add time for each data point and at times it might be advantageous to sacrifice accuracy for speed. The features of the different measurement types are compared to each other in the table below.

Table 2-1 Feature comparison for multi-point measurement types

| | **1-point** | **3-point** | **5-point** |
|---|---|---|---|
| reported moment | relative | absolute | absolute |
| compensation for linear drift in sample moment over measurement | no | yes | yes |
| compensation for linear drift in background moment over measurement | no | no | yes |

The software always assumes that the sample holder moment is uniform over the whole measurement range (about ±20mm from the sample center) and the entries in the table might not be accurate for non-uniform sample holders.

1-point measurements should only be used to quickly characterize sample properties (e.g., the presence and location of a transition) but the reported susceptibility and phase will never be completely accurate as they will include inhomogeneities of the sample holder (nulling is done in the “Z” position whereas the measurement is performed at the center position) and residual signals left after the nulling operation.

The preferred measurement uses the 3-point measurement which – due to the compensation of drifts in the sample moment – will also give accurate results for measurements while sweeping the temperature (unless the sample exhibits sharp and non-monotonic changes in its susceptibility). For samples whose susceptibility is small compared to the one of the sample holder, a 5-point measurement can give better results as it will compensate for drifts in the background/sample holder as well.

However, for very non-uniform sample holders, it might be advantageous to use a 1-point measurement as the inhomogeneity of the sample holder can result in the moment being reported incorrectly for multi-point measurements.

## 2.5.2 Optimizing Moment vs. Temperature and Moment vs. Field Sequence Commands

A few points should be kept in mind when writing sequences in order to get the most data in the shortest time. In this section a few general rules are listed and their reasoning is explained.

- **For sweeping measurements, use a single AC parameter set whenever possible**
  Changing either the drive amplitude or frequency between two AC measurements requires the software to complete another nulling command, which – especially at lower frequencies – can take longer than the actual data acquisition for the measurement. When measuring while sweeping temperature, this will result in potentially large gaps between the individual data points.

  When a single set of AC parameters is used, the software will issue a single nulling command at the very beginning, allowing to collect data much more quickly, resulting in larger data density. Moreover, the software optimizes the calculation of subsequent multi-point measurements using the same parameters (e.g., using a "C-T-C-T-C" measurement sequence rather than "C-T-C-C-T-C" to acquire two three-point measurements) which results in an additional increase in data density.

- **Use multiple loops in temperature or field rather than a single loop with multiple AC parameters**
  As temperature control is generally much faster than data acquisition when taking into account the additional time required for nulling, it is typically much faster to run multiple temperature sweeps using a single set of parameters for each rather than running a (much slower) single sweep with multiple parameters in order to get the same amount of data. This of course assumes that there are no history-dependent effects for the sample being investigated.

- **Use multiple repeat measurements at each stable field to investigate potential relaxation effects**
  When measuring individual data points at stable temperature or field, using the "Repetitions at each Field/Temperature" setting in the "Setup" tab of the Moment vs. Field/Temperature allows to collect multiple data points with the requested settings at each point, which allows investigation of time-dependent (relaxation) effects.

  When acquiring both VSM and AC data, the software will alternate between the two measurements (2 AC data points followed by 2 VSM data points) in order to minimize time spent to start and stop vibration of the linear motor for the VSM measurements. When using a single data point at each field or temperature, this will result in the order of measurements (VSM before of after AC) alternating between each set point. Using multiple repetitions at each set point however will still allow to study relaxation effects in both, VSM and AC data sets.

C H A P T E R 3

# Troubleshooting

## 3.1 Introduction

- Section 3.2 describes basic troubleshooting procedures for the AC Option

## 3.2 Troubleshooting

### 3.2.1 Verification of System Calibration

The SVSM AC Option is fully factory calibrated and should not require any recalibration by the user. For the original system calibration the supplied standard sample (ErYAG) was used which allows to verify the calibration at any time. Independent of temperature and frequency, the supplied standard sample should report an AC phase of zero and an AC Susceptibility that matches the slope of a dc (VSM) MvsH measurement between ±10 Oe.

Please contact your local Quantum Design representative for recalibration instructions if there is reason to assume that the calibration on the AC Option needs to be changed.

### 3.2.2 Sample Geometry Effects

Multi-point measurements use the gradiometer response as function of position to report the moment. As there are slight variations for the response for different sample geometries, the reported moment will only be correct for sample of the approximate size and shape of the standard sample used for the original calibration. For samples with a significantly different geometry, the reported moment will have a geometry-dependent correction factor. This factor can be measured by calibrating against a reference sample of known susceptibility with the same geometry as the sample being investigated.

A P P E N D I X A

# AC Data File

## A.1 Description of additional AC Data Columns

For systems with the AC Option installed, the measurement data file contains additional columns (both the VSM and AC data are collected in the same data file). These additional columns are present as soon as the option is installed, regardless of whether or not a measurement collects AC data. All data columns related to AC measurements start with "AC " in their name. Table A-1 explains the meaning of the additional data columns generated by the AC Option.

Table A-1 AC Option data columns

| Column | Description |
| --- | --- |
| AC Moment [a]<br>AC M. Std. Err. [a] | Amplitude of AC moment and associated standard error<br>This is the absolute value of the AC moment and will be always a positive number. Standard errors are only available when data has been collected for at least two cycles |
| AC Phase (deg)<br>AC Phase Std Err. (deg) | Phase (relative to the AC field) of the AC moment and associated error<br>Reported phase is in the range -90° to 270°, a pure in-phase signal will have a reported phase of 0° |
| AC Susceptibility [b]<br>AC Suscept. Std Err. [b] | Amplitude of AC susceptibility and associated standard error<br>Value is computed as *AC Moment/AC Drive* |
| AC X' [b]<br>AC X'' [b]<br>AC X' Std Err. [b]<br>AC X'' Std Err. [b] | In-phase and out-of-phase components of the AC Susceptibility and associated standard errors<br>These values can be calculated from the above AC Susceptibility and AC Phase and are provided for convenience |
| AC Drive [c] | Actual (peak) AC field applied to the sample<br>Field takes into account the total field generated from the modulation and nulling coils |
| AC Frequency (Hz) | Actual applied AC frequency<br>Value can be slightly different from requested value due to frequency resolution of the AC module |

| Column | Description |
|---|---|
| AC Averaging Time (s) | Actual averaging time used<br>Value can be slightly different from requested value in order to accommodate an integer number of cycles. For multi-point measurements, the averaging time represents the averaging time for each individual data point |
| **Diagnostic Items** | |
| AC Cycles | Number of cycles (individual waveforms) collected<br>For multi-point measurements, this is the minimum number of cycles of all the measurements. Value can be lower than requested by the user due to automatic rejection of bad cycles by the option |
| AC Range | SQUID range (1,10,100,1000) used for the data point<br>For multi-point measurements, this is the least sensitive range (largest number) of all the measurements |
| AC Measure Type | Integer representing the AC measurement type of the current data point<br>Possible values:<br>1 1-point measurement<br>3 3-point measurement<br>5 5-point measurement |
| AC Signal' (V)<br>AC Signal'' (V) | Raw SQUID signal (in-phase and out-of-phase), corrected for the SQUID range<br>Note that these values don't contain any phase and amplitude correction and should thus never be used for data analysis |
| AC Trim Coil Ratio<br>AC Trim Coil Phase | Amplitude ratio and relative phase of nulling field with respect to main AC field |

[a] Units depend on user selection – "emu" or "$Am^2$" based on selection in "Measure" menu

[b] Units depend on user selection – "emu/Oe" or "$Am^2/T$" based on selection in "Measure" menu

[c] Units depend on user selection – "Oe" or "T" based on selection in "Measure" menu

A P P E N D I X B

# Ordering Replacement Parts

## B.1 Overview

In this appendix we list the Quantum Design part number for loose or consumable parts you may wish to order throughout the lifetime of the SVSM AC Option. Contact your local Quantum Design representative to order parts.

| **Picture** | **Name** | **Part Number** |
|---|---|---|
|  | ErYAG standard sample | 4500-636 |