

# AC Susceptibility for Dilution Refrigerator

User's Manual



Quantum Design

# Additional Resources

This user manual has been compiled as a comprehensive reference to explain the theory, operation, and maintenance of your Quantum Design instrumentation. Additional materials related to this product may also be available on Pharos (Quantum Design's online repository for users) including representative data sets, example sequences, software utilities, etc. Video demonstrations and tutorials are available on Quantum Design's YouTube channel, and further documentation describing solutions to commonly encountered challenges in data collection and interpretation can be found in various Application Notes in the Applications section of the QDUSA website.



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For guidance regarding experimental design or data interpretation, as well as questions regarding the content of this manual, please reach out to the Applications group.

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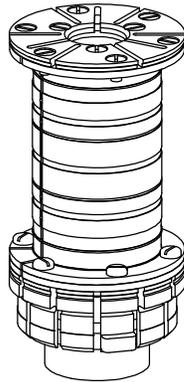
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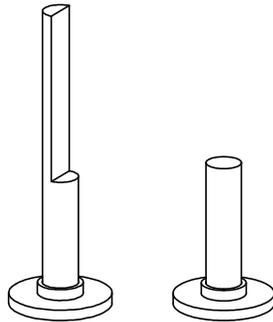


# Key Components



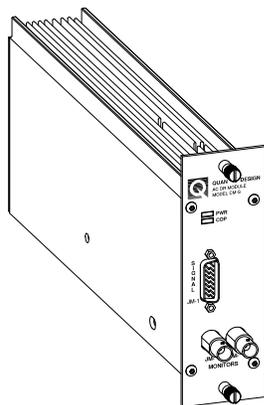
## ACDR Coil Set

Comprises the drive, detection, trim, and calibration coils employed by the measurement.



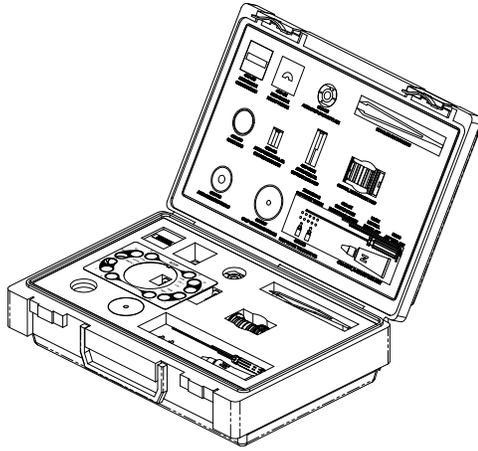
## Sample Holders

Thermally connects the sample to the DR mixing chamber; planar samples are easily oriented with the applied DC/AC magnetic fields in-plane (left), or out-of-plane (right).



## ACDR CAN Module

Contains electronics that source excitation drive and trim currents; detects voltages induced by sample response.



## User Kit

Contains various accessories to facilitate proper use of the measurement option, including:

- GGG Standard Sample (2 pcs., 4091-609)
- Sample mount plate (4091-612)
- Radiation Shield (4091-706)
- Sample Height Fixture (4091-621)
- Sample Centering Fixture (4091-707)

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## Overview of the ACDR

### 1.1 What the Option Measures

The AC Susceptibility for Dilution Refrigerator (ACDR) Option enables measurements of the dynamic susceptibility of samples within the low temperature regime of the Dilution Refrigerator Option for the PPMS (0.05 K – 4 K). The sample is mounted on a sapphire rod anchored to the mixing chamber of the DR probe allowing the sample to be cooled. The same rod, when the DR is installed, sits concentrically within the ACDR coil set (itself thermally decoupled from the DR and remains at or near the base temperature of the PPMS). The coil set provides the oscillating AC magnetic field excitation at a user-selected frequency (between 10 Hz – 10,000 Hz) and amplitude (up to 4 Oe) and also detects the oscillating response of the sample. Let's add more words.

A lock-in amplifier-based technique extracts the amplitudes of the in- and out-of-phase components (as compared to the drive signal) of the induced voltage in the detection coils. This voltage can be related to the dynamic susceptibility through calibration constants (provided upon installation by Quantum Design, but verifiable by the user at any time using the included GGG reference sample) and is reported to the user.

Though the fundamental ACDR measurement technique is essentially unchanged from the AC Measurement System (ACMS-II) Option for the PPMS, the mechanical limitations and space constraints of the DR make multi-point measurements impossible. As a result, there is an additional step required (nulling) to remove spurious background signals and ensure the reported signal is as accurate as possible.

Finally: this manual presumes the user already has some familiarity and experience with the Dilution Refrigerator. For users new to DR measurements, first read thoroughly the Dilution Refrigerator Option User's Manual before proceeding to attempt any ACDR measurements.

### 1.2 Theory of Operation

The ACDR Option is designed as an automated, turn-key system for measuring the dynamic susceptibility of a material at DR temperatures. As such, users need not be experts on the topics of susceptometer construction and lock-in amplifiers to successfully use the system and acquire high-quality data. With that said, frequent users of the ACDR, or those who are simply curious, may benefit from a more detailed understanding of the principals at work behind the measurement.

These proceeding topics are best revisited in detail when the user already has a passing understanding of the measurement and should not be considered prerequisite knowledge for operating the ACDR.

#### 1.2.1 Origins of the AC Susceptibility Signal

Consider the ACDR coil set's detection coil: a pair of counter-wound turns with the same radii (each numbering  $n$ ) of a single metallic wire centered in the PPMS sample chamber, where the two sets of turns are displaced by a small amount along the axial direction. When the DC field is applied the flux through each set of turns has an equal and opposite sign, so nominally the total flux from the DC field is zero. Once the sample, affixed to the DR, is installed, it resides at the center of one of these sets of turns; in this way, one set of turns sees a great deal more flux from the

sample than does the other. This design is thus sensitive to the magnetic field originating from the sample, but not that of either the applied constant DC field, or the small uniform AC excitation field which are superimposed.

Next examine the case of an excitation field at a very low frequency (e.g., 0.01 Hz), where the measurement is functionally equivalent to DC magnetometry. Here, the magnetic moment of the sample faithfully follows the  $m(H)$  curve that would be measured in a DC experiment, only now the applied field is the time-dependent  $H = H_{AC} \sin(\omega t)$ . For this limiting case, it is useful to ask, "What is the resulting voltage induced in the detection coil?" Starting from Faraday's Law:

$$\mathcal{E} = -\frac{d\Phi}{dt}$$

...where the magnetic flux  $\Phi$  can be understood as the surface integral (integrated over two surfaces, each bounded by one set of turns of the detection coil) of the magnetic field produced due to the presence of a magnetic dipole centered at one of the sets of turns:

$$\Phi = n \left[ \iint \mathbf{B}(\mathbf{r}, \mathbf{m}) \cdot d\mathbf{A}_1 - \iint \mathbf{B}(\mathbf{r}, \mathbf{m}) \cdot d\mathbf{A}_2 \right]$$

A few points to note about the above expression: First, to reiterate, the two integrals are executed over different open surfaces ( $\mathbf{A}_1, \mathbf{A}_2$ ) and they carry opposite signs- due to the two sets of  $n$  turns being counter-wound. Second, the magnetic field due to the dipole depends both on the size  $m$  and direction  $\hat{m}$  of the moment vector  $\mathbf{m}$ , but also the position in space  $\mathbf{r}$  for the various points across the surfaces bounded by the turns of wire. It can be shown that the *magnitude* of the magnetic field from a dipole is linear in the magnitude  $m$  of the moment vector, which allows this expression to be re-written as:

$$\Phi = m \cdot \left\{ n \left[ \iint \mathcal{B}(\mathbf{r}, \hat{m}) \cdot d\mathbf{A}_1 - \iint \mathcal{B}(\mathbf{r}, \hat{m}) \cdot d\mathbf{A}_2 \right] \right\}$$

...where naturally  $\mathcal{B} = \mathbf{B}(\mathbf{r}, \mathbf{m})/m$ , thus reducing the integration terms to a problem of geometry. Once its solution is found, the braced terms will be constant for any sample, so it is simply labeled  $C$ . This substantially simplifies the expression for induced voltage:

$$\mathcal{E} = -C \frac{\partial m}{\partial H} \cdot \frac{dH}{dt}$$

$$\mathcal{E} = -C \frac{\partial m}{\partial H} \cdot H_{AC} \cos(\omega t)$$

Since the constant  $C$  can be calculated from the coil geometry, and both the excitation field amplitude  $H_{AC}$  as well as the angular frequency  $\omega$  are known, it is thus shown that the induced voltage, for low frequency excitations of a non-hysteretic material, is fundamentally a measure of the *slope* of the  $m(H)$  curve,  $\partial m / \partial H$ .

This is perhaps the most important and distinguishing property of AC magnetometry, standing in contrast with a DC experiment where  $m$  itself is the property directly measured. One advantage of this technique is now immediately evident: since the AC measurement is sensitive not to the moment value but instead to the slope of  $m(H)$ , small changes in the moment can be detected much more easily, even when the absolute value is large.<sup>1</sup>

At higher frequencies more typical of an AC measurement (100's or 1000's of Hz or more), the AC moment of the sample does not generally match the slope of the DC magnetization curve due to dynamic effects in the sample.

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<sup>1</sup> A second, subtler point to note is that the induced voltage response is 90° out-of-phase from the AC drive current/field [ $H_{AC} \propto \sin(\omega t)$ , while  $\mathcal{E} \propto \cos(\omega t)$ ], so the *real* component of the AC moment/susceptibility is actually calculated from the voltage component which is *out-of-phase* with the AC excitation field.

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. The fundamentally measured quantities are the in-phase (‘real’) component  $X'$  and an out-of-phase (‘imaginary’) component  $X''$ , but these can also be represented as a phasor having a distinct magnitude  $X$  and phase angle  $\phi$  (relative to the drive signal).

The two representations are related by the following equalities:

$$\begin{aligned} X' &= X \cos \phi & X'' &= X \sin \phi \\ X &= \sqrt{X'^2 + X''^2} & \phi &= \text{atan2}(X'', X') \end{aligned}$$

...where **atan2()** is the two-argument atan2 function.<sup>2</sup>

In the low frequency limit discussed prior, the real component  $X'$  is just the local slope of the  $m(H)$  curve, and the imaginary component  $X''$  (which indicates the presence of dissipative processes in the sample) should be near zero. For samples where some loss mechanism introduces hysteresis in the  $m(H)$  curve, such as a ferromagnet, there can be significant signal in the  $X''$  channel. Both  $X'$  and  $X''$  are very sensitive to thermodynamic phase changes and are often used to identify precise transition temperatures.

## 1.2.2 Measured Quantities

A sample’s true dynamic (AC) susceptibility has real (in-phase) and imaginary (out-of-phase) components to its response, indicated by  $\chi'$  and  $\chi''$  respectively (this notation, lacking a subscript, typically implies the volume susceptibility [1]). It is important to note that the ACDR options *does not* report this volume susceptibility strictly speaking, but instead the real and imaginary components of  $\partial m / \partial H$  as described in [Section 1.2.1](#). This quantity is represented in this manual and in the ACDR software by the Latin character  $X$ . For an AC measurement, the volume susceptibility  $\chi$  and reported quantity  $X$  are related by:

$$\begin{aligned} \chi' &= \text{Re} \left[ \frac{\partial M}{\partial H} \right] = \frac{1}{V} \cdot \text{Re} \left[ \frac{\partial m}{\partial H} \right] = \frac{1}{V} \cdot X' \\ \chi'' &= \text{Im} \left[ \frac{\partial M}{\partial H} \right] = \frac{1}{V} \cdot \text{Im} \left[ \frac{\partial m}{\partial H} \right] = \frac{1}{V} \cdot X'' \end{aligned}$$

...where the volume magnetization is indicated by the typical symbol  $M$  and is related to the net magnetic moment of the sample  $m$  by:

$$M = \frac{m}{V}$$

Since a precise measure of the volume is often impractical for samples of the size usually employed for an ACDR measurement, often a more convenient intrinsic quantity used is the mass susceptibility  $\chi'_\rho$  :

$$\chi'_\rho = \frac{1}{\rho} \cdot \chi' = \frac{1}{b} \cdot X'$$

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<sup>2</sup> **atan2**( $y, x$ ) =  $\tan^{-1}(y/x)$  for  $x > 0$  ; **atan2**( $y, x$ ) =  $\tan^{-1}(y/x) + 180^\circ$  for  $x < 0$  .

[1] R. B. Goldfarb and F. R. Fickett, "Units for Magnetic Properties - NBS Special Publication 696," U.S. Department of Commerce, National Bureau of Standards, Boulder, CO, 1985.

...where the sample density is  $\rho$  and its mass is  $b$ , and the analogous form is implied for  $\chi''_{\rho}$ .

This is mentioned here to stress the relation between the *extrinsic* quantity reported by the ACDR Option-  $X'$ , and the intrinsic quantities most often of interest for purposes of comparison or use in further calculations such as  $\chi'$  and  $\chi'_{\rho}$ . Outside of this discussion, the manual will exclusively use  $X$ , which may be referred to colloquially as 'the susceptibility', but the meaning is always the one described explicitly here. A simple scaling by volume, mass, or number of moles will still generate the corresponding intrinsic values.

### 1.2.2.1 Reported Units

The units of the physical quantities discussed prior are summarized in Table 1-1. As with all Quantum Design measurement options dealing with magnetic properties the default unit system is the 'electromagnetic units' (EMU) variant of CGS.

**Table 1-1:** Reported units of select properties.

Property Name	Units	Description
$X, X', X''$	emu/Oe	Susceptibility
$\phi$	degrees	Phase
$H_{DC}$	Oe	Applied field
$H_{AC}$	Oe	Excitation field amplitude
$f$	Hz	Excitation frequency

Note that the units of the reported quantities are also always indicated by the column titles within header of the .dat file as well.

### 1.2.2.2 Converting to SI

With the increasing prevalence of SI units in studies of magnetism, it is helpful to know the proper conversion factors to transform the EMU CGS units reported by the ACDR Option to their equivalent units in SI. Such a conversion is further complicated by the choice of the designator 'emu' for the units of the magnetic moment  $m$ . 'EMU' simply refers to the chosen *subsystem* of CGS used to quantify magnetic properties; other examples include the 'electrostatic units' (ESU), Gaussian, and Lorentz-Heaviside system[2], though none of these are employed in Quantum Design options.

The dimensionality of a magnetic dipole moment  $m$  is current times length-squared [ $I \cdot L^2$ ]; in the EMU CGS system, the unit for current is the abampere [ $abA$ ], which is also called a biot<sup>3</sup> [ $Bi$ ], and the unit for length is of course the centimeter [ $cm$ ]. It follows then that the units for a magnetic dipole moment are [ $Bi \cdot cm^2$ ] [3]. It is *this* quantity to which the designator 'emu' is referring when encountered as a pseudo-unit in Quantum Design measurement options.

With that now understood, Table 1-2 provides the relevant conversion factors for translating the constituent and derived units of the reported quantities into SI. The conversation is defined such that multiplying a value in the 'Reported Units' by the conversion factor will a result in the proper magnitude for the same quantity in 'SI Units'.

[2] J. D. Jackson, Classical Electrodynamics, 3rd Ed., New York: Wiley, 1998.

<sup>3</sup> In honor of the French physicist Jean-Baptiste Biot, of Biot-Savart Law fame.

[3] F. Cardarelli, Encyclopaedia of Scientific Units, Weights and Measures, London: Springer, 2005.

**Table 1-2:** Conversion factors for select units of magnetic properties.

Quantity	Symbol	Reported Units	Conversion Factor	SI Units	Notes
Magnetic Moment	$m$	[Bi · cm <sup>2</sup> ]	10 <sup>-3</sup>	[A · m <sup>2</sup> ]	Labeled as 'emu'
Magnetic Field	$H$	[Oe]	$\frac{10^3}{4\pi}$	[A · m <sup>-1</sup> ]	
Susceptibility	$X, X', X''$	$\frac{[\text{Bi} \cdot \text{cm}^2]}{[\text{Oe}]}$	$10^{-3} \cdot \left(\frac{4\pi}{10^3}\right)$	$m^3$	Not the true volume susceptibility, $\chi$

### 1.2.3 Nulling the Coil Set

A key assumption made in some of the earlier analysis is that the signal detected by the ACDR Option derives purely from the presence of the user's sample within the coil set, which is not necessarily the case. Any number of other sources can cause spurious signals to arise in the detection coils, including (but certainly not limited to):

- **Imperfect detection coil windings:** As a macroscopic object there are limits to the degree of symmetry achievable between the two sets of turns comprising the detection coil. This imbalance, though small, results in the oscillating excitation field not being fully canceled, which in turn induces an additional voltage response in the detection coil.
- **Imperfect drive coil windings:** Similar to the situation described above, slight non-uniformities in the drive coil can produce small asymmetries in the field profile. This means the excitation field is not perfectly canceled by the first-order gradiometer and can be considered another source of induced voltage in the detection circuit.
- **Sample holder itself:** Since the material comprising the holder sits inside the detection coil as well, its own response is measured and needs to be accounted for. The degree to which the holder couples to the detection coils is sensitive to the precise spatial location of the holder. As a result, it is necessary to re-null every time the sample holder is removed to account for small shifts in position due to remounting.

Engineering constraints prevent the cited issues from being completely removed from the sample measurement, so the next best option is to carefully quantify their contribution. This quantification is acquired during a measurement of the empty sample holder. As a first step, the software measures the empty sample holder at various excitation amplitudes and frequencies to quantify the total background measurement. Next, current is supplied, based on the prior result, to the trim coils in order to cancel (null) the measured background. The measurement is repeated with the trim coils energized to confirm they have properly canceled the background signal.

When the proper parameters for successful nulling have been determined for the full excitation amplitude and frequency space they are written to the `ACDRCOIL-000.cfg` file (where 000 corresponds to the actual serial number of the user's coil set). With the nulling parameters recorded, for every measurement of the sample the relevant excitation parameters are applied to the trim coil to actively null the background signal. This way, any remaining signal can be reasonably inferred to originate from the sample alone.

If possible, it is best to perform the nulling at the same frequencies that will be used during the experiment. A simple linear interpolation of the nulling parameters is used when measuring between frequencies. More information is provided in [Section 4.1.1.7](#) regarding the default frequencies used during nulling and how to change them.

This drastically improves the accuracy of the ACDR, though it is still not as sophisticated or effective as the multi-point measurements of the ACMS-II Option, where the sample can be physically translated to different points along the axis of the coil set. If further improvements to the measurement accuracy are required, refer to [Application Note 1091-217](#), *Assigning an Absolute Scale to Susceptibility Measurements with the ACDR*.

## Hardware Descriptions

### 2.1 The ACDR Coil Set

The coil set contains several different coils of wire in various configurations, each of which plays a crucial role in the AC susceptibility measurement. All the coils are concentrically wound and positioned about the central axis of the PPMS sample chamber- this is also the center of the superconducting magnet providing the constant DC magnetic field. The roles of these different coils are described subsequently.

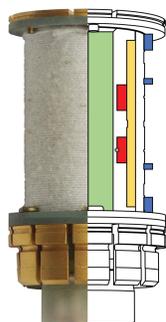
#### 2.1.1 Drive Coil

The drive coil is a thin superconducting wire wound longitudinally around the detection coil set. The drive coil can generate alternating fields of up to 4 Oe spanning a frequency range of 10 Hz to 10 kHz. Constructing the drive coil from superconducting wire minimizes the heat generated when applying AC fields and allows the option to work down to the lowest temperatures with virtually no impact on the DR cooling power.

When measuring at the lowest temperatures, the maximum AC drive amplitude may need to be reduced at the highest frequencies to avoid heating of the sample or the coil set by inducing eddy currents. Carefully monitor the DR and puck temperatures and adjust the measurement parameters as needed. Additionally, the composition of the superconducting wire used in the drive coils limits the maximum DC field to  $\pm 12$  T; larger fields will quench the drive coils and cause excessive heating.

#### 2.1.2 Compensation Coil

A compensation coil is situated outside the AC drive coil. The drive coil and compensation coil are counter-wound and connected in series so that they receive the same excitation signal. A net field remains within the measurement region, but outside the measurement region the fields from the two coils cancel. This design effectively confines the excitation fields to the volume of the coil set, thereby reducing interactions with the conductive materials outside the measurement region (e.g., the sample chamber walls, the DR radiation shield, and sample stage). This drastically reduces eddy current heating and enables AC measurements to be performed even at the highest frequencies and lowest sample temperatures.



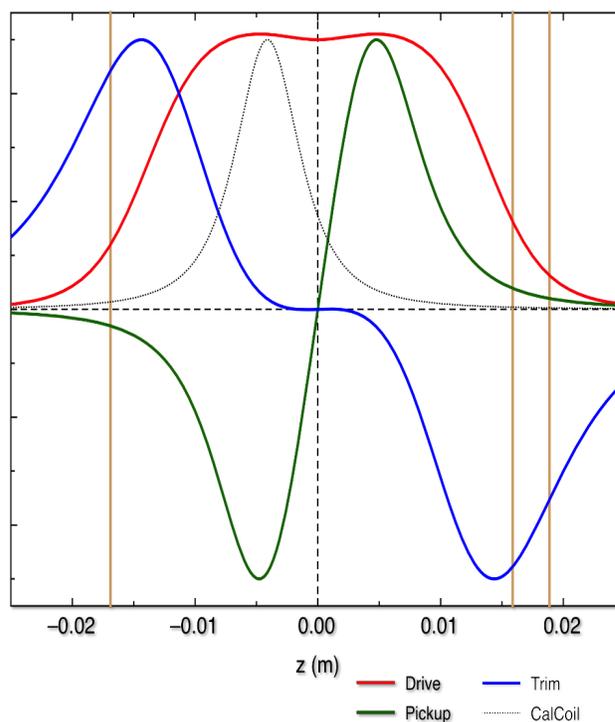
**Figure 2-1:** Schematic representation of the ACDR coil set. The bore for the sample holder is shown in green; also shown are the drive coil (yellow), detection coil (red), trim coil (blue), and calibration coil (gray). The compensation coil is not depicted for clarity.

The detection coils are counter-wound copper coils arranged in a first-order gradiometer configuration to help isolate the sample's signal from the uniform background field. During AC measurements, an alternating field is applied to the measurement region in superposition to the DC field of the magnet, while the sample is located in the center of the upper detection coil. The signal from the detection coil reflects the changes in the magnetization of the sample, due to the excitation field, and is processed by the lock-in detection electronics in the module.

### 2.1.3 Trim Coil

The trim coil is made of multiple turns wound in such a way that it has a large mutual inductance with the detection coil while still not contributing any field at the sample location (see Figure 2-2). Applying current to the trim coil, due to the coupling with the detection coil, allows for the frequency dependent background signals measured during the nulling procedure to be actively canceled out from the signal measured in the detection coil. This can be accomplished without otherwise perturbing the sample during a measurement due to the aforementioned geometry. The use of the trim coil to null the background allows the system to be able to report more accurate absolute susceptibility values.

**Figure 2-2:** Field profiles of various coils in the ACDR coil set. The z coordinate is centered at the midpoint between the two sets of turns comprising the detection coil (green). Note the minimal coupling between the detection (green) and trim (blue) coils at the sample location near +0.5 cm, and the calibration coils position at -0.5 cm. The drive coil (red) is wound such that its profile is relatively uniform across the length of the detection coil. The gold lines represent metallic surfaces corresponding to, from left to right: the surface of the puck upon which the coil set is mounted, the surface of the DR radiation shield, and the surface of the plate used to mount the stand-off to the DR stage.



### 2.1.4 Calibration Coil

The bottom detection coil also contains a single-turn low-inductance calibration coil situated at the center of the bottom detection coil. Since the calibration coil can provide a known AC susceptibility signal, on-the-fly measurements of any attenuation and phase shift introduced by the measurement system can be quantified and corrected.

In light of the resistive and inductive character of the coils, all AC measurements will present phase shifts between the drive and the detected signals. These phase shifts can be dependent on temperature as well as the AC drive frequency, and therefore are susceptible to changes across the parameter space of a given measurement. The calibration coil aids in being able to separate the true sample phase shift from instrumental shifts, allowing for accurate reporting of phase angles to better than  $\pm 2^\circ$ .

## 2.2 Sample Thermometry

In the ACDR Option samples are mounted on a thermal stand-off attached to the sample stage of the DR; the temperature of the DR stage can be accurately controlled between 50 mK and 4 K. The ACDR coil set remains at a

constant temperature of 1.8 K or 2.5 K depending on the cooling mode of operation (i.e., dilution or evaporative cooling respectively). This design provides fast thermal response times due to the limited thermal mass added to the DR and reduces heat load from the AC drive current onto the sample stage.

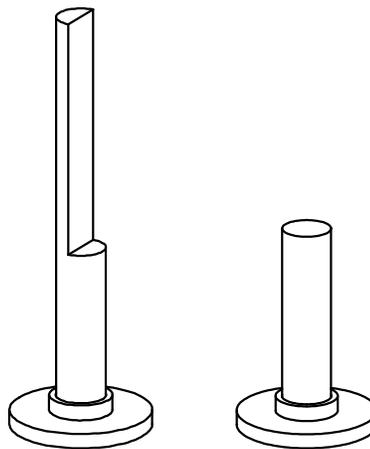
While this design allows AC measurements to be performed down to 50 mK, the setpoint of the DR stage is not necessarily representative of the true sample of the temperature. This is due to the finite thermal conductance of the sapphire standoff and as a result the sample temperature can lag the DR setpoint. This is particularly an issue when performing measurements below 100 mK. Direct thermometry measurements of the end of the stand-off indicate that equilibration times can be on the order of ten minutes in this regime. If the sample has a known temperature dependence, the AC magnetometry signal from the sample itself can be used to monitor temperature, otherwise a liberal allowance of time for thermal equilibration is strongly recommended.

## 2.3 Sample Holders

Two stand-offs are provided which each afford a flat surface to mount a user's sample (shown in [Figure 2-3](#)). The single crystal sapphire material is chosen as it provides superior thermal conduction at low temperature, mitigating the thermal lag between the DR stage and the sample itself. A small gold-plated silver fixture (also chosen for its thermal conductance) holds the stand-off to the sample stage of the DR, which is in direct contact with the mixing chamber.

The functional difference between the holders is that one makes it easy to mount a planar sample with the static and excitation fields normal to the plane of the sample ('perpendicular') and the other positions such a sample with the fields in the plane of the sample ('parallel'). For materials with an isotropic response, where the orientation is not important, the minor advantage of the latter is that the sapphire material fully extends through both windings of the detection coils; this more uniform background is easier to null and can provide a slight improvement in data quality for low-signal materials.

**Figure 2-3:** Sample holders for the AC DR Option. At left is the 'parallel' holder, at right the 'perpendicular' one, both described prior. The static DC field and the AC excitation field are directed along the long axis of each holder.



## 2.4 ACDR Control Area Network (CAN) Module

The ACDR Module contains the electronics that produce the desired excitation current in the drive coils, and it also measures and separates the real and imaginary components of the sample's magnetic response from the signal produced in the detection coil. The heart of this module is a high-speed AC stimulus response (ACSR) card that synthesizes the excitation signal and processes the detection coils' response signal.

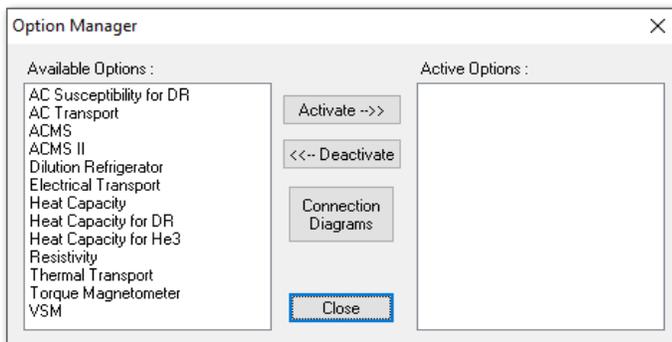
The ACDR coil set interfaces with the 12-pin puck-style connector located at the base of the PPMS sample chamber and is connected to the module (JM-1) through via the 14-pin LEMO socket at the top of the PPMS.

For the purposes of diagnostics, the module also features two BNCs which can be used to monitor the output of the detection coil after the amplification (JM-2) and the drive coil reference signal (JM-3).

### 3.1 Getting Started

The latest versions of both MultiVu and the ACDR Option software can always be found on the Quantum Design website.<sup>1</sup> Check occasionally to confirm the latest version is installed, as updates can contain additional features, ease-of-use improvements, or important bug fixes.

The ACDR Option software can be activated by browsing to the Option Manager via the menu items **Utilities>Activate Option...**, choosing 'AC Susceptibility for DR', and clicking **Activate**. The Dilution Refrigerator can be activated at the same time, in the same way.



**Figure 3-1:** Option Manager window.

### 3.2 Interface Walkthrough

When the ACDR Option is first activated, the main AC DR control window and the AC DR Log window both automatically appear. Their functions as well as that of the primary measurement dialog window are described below.

#### 3.2.1 AC DR Log Window

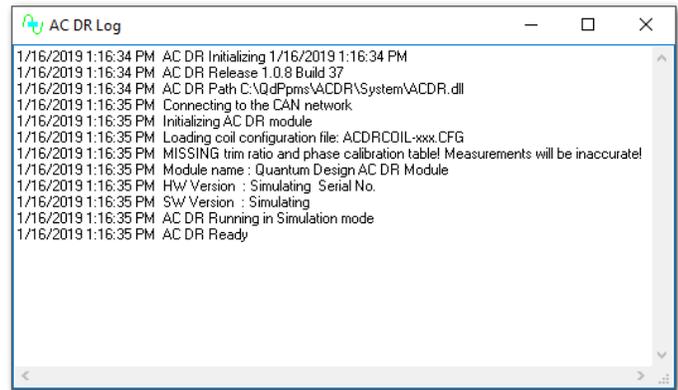
The AC DR Log window displays various status updates generated by the option software. As part of the initialization procedure, the present software version is declared, along with the file name of presently-loaded .cfg file, and communication with the module is initiated. If the software detects any errors, they are reported here.

---

<sup>1</sup> Under the 'Software Upgrades' section:

[https://www.qdusa.com/support/software\\_upgrades.html](https://www.qdusa.com/support/software_upgrades.html)

**Figure 3-2: AC DR Log window.**

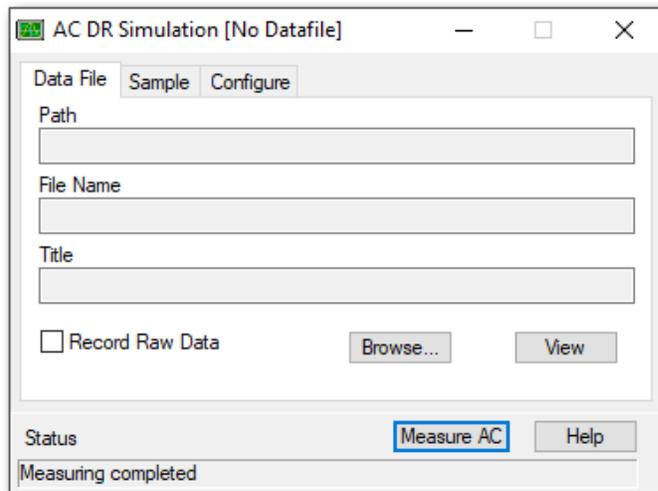


During a measurement, this log will continually record key events in an ACDR measurement (e.g., changing the data file, starting a measurement, etc.). Anything appearing in the AC DR Log window is also written to the `\ACDR\LogFiles\ACDRLog.txt` file in case it is needed for later review.

The AC DR Log window can always be recalled to the foreground with the menu command **View>AC DR Status Log**.

### 3.2.2 AC DR Window

The main window for controlling the option, shown in **Figure 3-3**, contains three main tabs (Data File, Settings, and Configure). **Measure AC** opens the AC DR Susceptibility dialog to configure and start an immediate measurement and is discussed further in Section 3.2.3. At the bottom of the window is a small status box which relates the most recent line from the AC DR Log window.



**Figure 3-3: Main AC DR window for option control.**

#### 3.2.2.1 Data File Tab

The Data File tab relates the file path and file name where acquired AC susceptibility data are stored. The **View** button will display a plot of the current file, while the **Browse...** button can be used to choose a different file or start a new one. If creating a new file, the user is prompted to choose a title for the file and given the chance to record relevant sample parameters; this dialog is shown in **Figure 3-4**.

While all of these dialogs are optional (they can be left blank) it is recommended that any unique identifiers of the sample, as well as parameters needed for scaling (such as mass) be recorded at this time. All of these fields are written into the header of the data file; if further analysis is required at a later time, this ensures that the data can be properly related to the correct sample and correctly scaled after the fact, using only the `.dat` file.

**Figure 3-4:** AC DR Sample Properties window.

The screenshot shows a dialog box titled "AC DR Sample Properties" with a close button (X) in the top right corner. Inside the dialog, there is a question mark icon and the text "Enter Sample Properties". Below this, there are several input fields: "Material", "Comment", and "Additional Descriptive Comments". Under "Additional Descriptive Comments", there are four more input fields: "Mass" (with "mg" next to it), "Size", "Volume" (with "mm<sup>3</sup>" next to it), and "Shape". At the bottom of the dialog, there are three buttons: "OK", "Cancel", and "Help". The "Cancel" button is highlighted with a blue border.

The **Record Raw Data** checkbox can be selected to retain in a `.raw` file certain lower-level information used to generate the results reported in the `.dat` file. These include the in- and out-of-phase voltage signals, drive field amplitude and frequency, among other parameters; for a full listing and brief descriptions, see [Section 3.4.2](#). It is important to note: *no raw waveform data* is stored in this file. The lock-in technique used extracts the average amplitudes of the real and imaginary parts of the harmonic component of the measured voltage signal at the frequency matching that of the drive frequency  $\omega_0$ . The raw response  $V(\omega, t)$  containing all harmonic content is not recorded.

### 3.2.2.2 Sample Tab

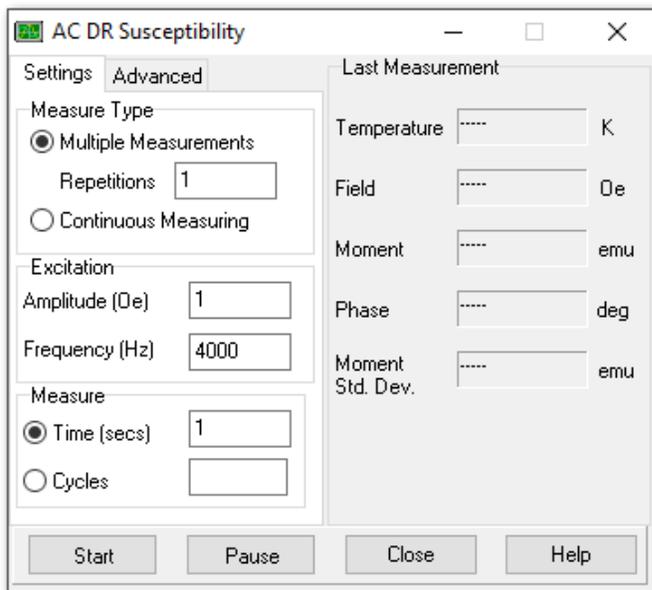
This tab provides a convenient way to view the sample properties of the presently active data file. This information cannot be edited once a `.dat` file is created; if a new sample is installed or the sample is otherwise modified, create a new `.dat` file as described in [Section 3.2.2.1](#).

### 3.2.2.3 Configure Tab

The button on this tab, **Change Coil Configuration**, allows the user to specify a new coil serial number and the ACDR Option software will load the associated `.cfg` file if present in the `\ACDR\Calibration\` folder. After pressing **Next**, the results of loading the new configuration will be recorded in the AC DR Log window.

## 3.2.3 AC DR Susceptibility Dialog

This window contains the main available set of options used to configure AC susceptibility measurements using the ACDR Option and can be used to trigger an immediate measurement. It is accessed either by the **Measure AC** button in the AC DR main control window, or via the **Measure>AC DR Susceptibility...** menu item. This window is often useful for quick tests to determine the appropriate settings within the parameter space of excitation field amplitudes and frequencies, whereas more systematic measures of AC susceptibility as functions of temperature or applied DC field are typically configured with sequence commands, described later in [Section 3.3.1](#).



**Figure 3-5:** AC DR Susceptibility window showing the Settings tab.

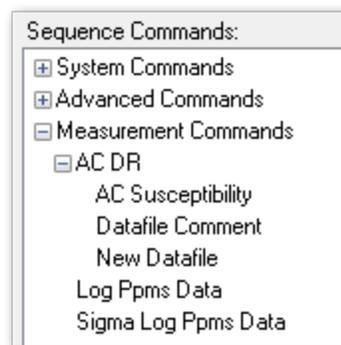
Under the **Measurement Type** frame users can direct the system to perform an integer number of **Multiple Measurements** before the system will stop measuring, or **Continuous Measuring**, which after initiated will collect data until some intervention by the user. The Excitation frame allows the Amplitude of the sinusoidal magnetic field excitation to be specified with a maximum limit of 4.4 Oe. The Frequency of the excitation must fall in the range from 10 Hz to 10 kHz. Finally, the length of the measurement is specified, either in **Time (seconds)**, or the number of **Cycles (periods)** across which the system executes the lock-in measurement.

The other panel of the window reports the result of the most recently completed AC susceptibility measurement. Buttons along the bottom allow the user to **Start** or **Pause** a measurement (the first button turns to **Stop** during an active measurement); one can also **Close** the AC DR Susceptibility dialog or access the **Help** resources from this panel.

### 3.3 Sequence Command List

Activating the ACDR Option software causes a new set of sequence commands to be added to the list shown in the **View>Sequence Command Bar** area under the Measurement Commands listing (shown in [Figure 3-6](#)).

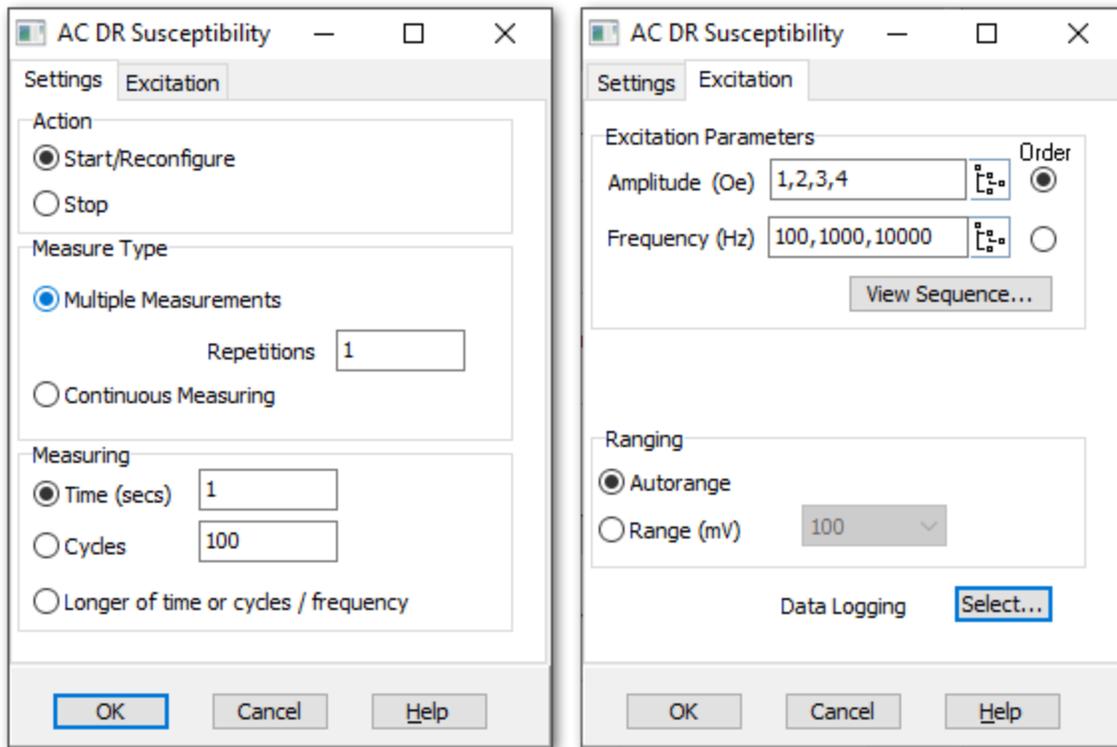
**Figure 3-6:** Available AC DR Option measurement commands listing.



The functionality of these commands is described individually in the order they are listed.

#### 3.3.1 AC Susceptibility

All of the measurement functionality of the ACDR Option resides within this single sequence command. For the most part, it replicates the functionality of [Section 3.2.3's](#) AC Susceptibility dialog, with a few additional features added to make systematic collection of data more straightforward.

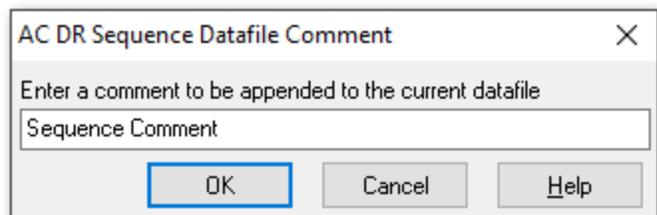


**Figure 3-7:** AC Susceptibility sequence command configuration window. Both the Settings and Excitation tabs are shown.

### 3.3.1.1 Settings Tab

The **Settings** tab, shown at left in [Figure 3-7](#), features both an **Action** frame and a **Measurement Type** frame; the selections here depend on the way in which the user would like to structure their sequence and measurement.

**Figure 3-8:** Datafile Comment sequence command dialog.



There are two strategies:

- a. The user includes a **Start** command using the **Continuous Measuring** selection, so that the AC susceptibility is constantly being measured. While this occurs, an external parameter can be varied at a set rate (temperature, field, or just waiting for some fixed time to elapse); when that is complete, a **Stop** command will halt the measurement. The advantage here is the measurement will finish in a known period of time, but the downside is the resulting point density cannot be easily predicted.<sup>2</sup>

<sup>2</sup> This issue is further complicated if the temperature range traverses 1.1 K, where the dilution refrigerator must change cooling modes; during this time the temperature changes too rapidly to guarantee sufficient thermal equilibration.

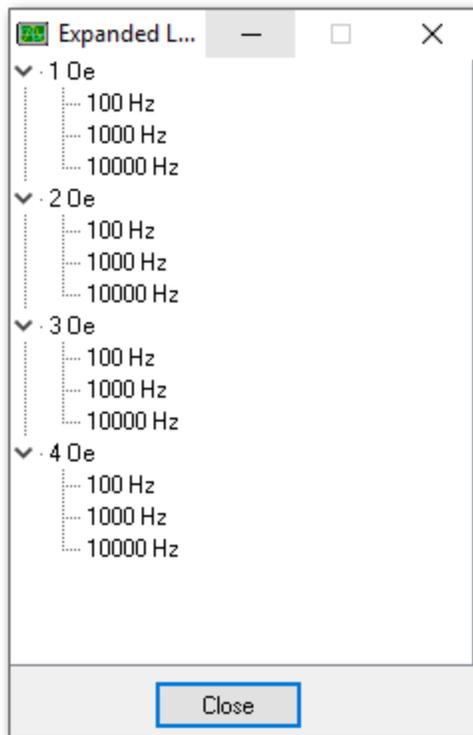
- b. The AC Susceptibility **Reconfigure** command (which is the same radio button as for **Start**) with the **Multiple Measurements** setting chosen is nested inside of a scan command (again, either temperature, field, or time). Here, measurement only executes at pre-determined points in the range of the external parameter. The situation is now the opposite as above: the time it will take the measurement to finish is unknown, but the point density is fixed by the parameters of the scan command. The **Repetitions** can be increased if the user would like multiple points collected at each step, typically for subsequent uncertainty analysis.

The last frame, **Measuring**, on this tab deals with the amount of time a single measurement (i.e., a 'repetition') consists of. The first choice is the most obvious: the system will measure for a fixed period of **Time (seconds)**.<sup>3</sup> The second allows for a fixed number of **Cycles** (oscillations of the drive field). The last option, **Longer of time or cycles / frequency** uses input from both parameters, and chooses the value that results in the longest measurement; this is best used when a wide range of frequencies will be sampled. With this selection, input the minimum number of cycles that would be acceptable for low frequencies (to avoid under-sampling) and the maximum amount of time the system should spend on measurements at high frequencies (to avoid wasting time collecting millions of cycles when a few thousand will do).

### 3.3.1.2 Excitation Tab

Shown at right in [Figure 3-7](#), the **Excitation Parameters** frame in the **Excitation** tab allows the user to define the points the amplitude/frequency parameter space to be sampled. Clicking the tree-structure icon opens a small dialog where the start and end values can be specified, as well as the number of points comprising the range ('count'). The spacing schemes for both the **Amplitude** (options: linear, log, inverse, square, square-root) and the **Frequency** (options: linear, log, inverse) can be specified, and the option software automatically calculates the set of amplitudes and frequencies. Arbitrary values can also be input manually, separated by a comma.

The **Order** radio button selects which set is a subset of the other, and the entire sequence can be previewed by using the View Sequence... button. An example preview can be seen in [Figure 3-8](#) and was generated from the values shown in the right panel of [Figure 3-7](#). Note that only a single excitation amplitude and frequency pair may be selected if the measurement is being operated in continuously, as described in [Section 3.3.1.1](#).



**Figure 3-9:** Preview of excitation parameter measurement ordering. In this example, 'order' was assigned to the amplitude.

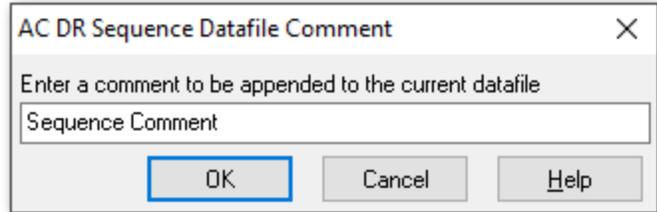
<sup>3</sup> Or rather, as near to the specified time as is possible, given that the cycle period will generally not divide evenly into an arbitrarily specified length of time: e.g., a time of 1.5 seconds for a frequency (period) of 13 Hz (0.077 s) will only accommodate 19 full cycles.

The bottom frame allows the user to determine the **Ranging** scheme; the default of automatic ranging is typically used, but advanced users may prefer to set the range manually. **Data Logging** can be used if MultiVu was earlier configured to record some external parameter.

### 3.3.2 Datafile Comment

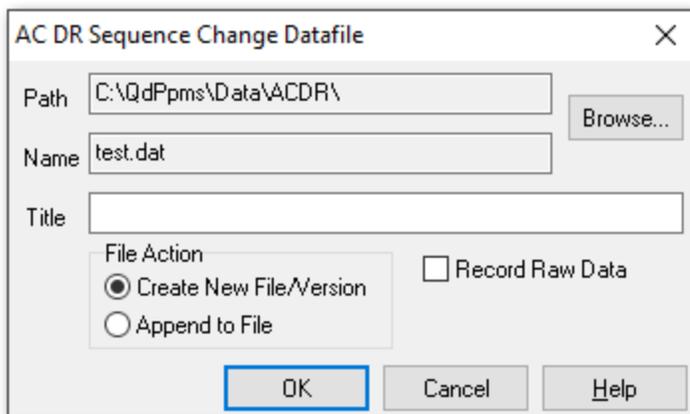
If it is helpful to include some notes or additional information in-line with the AC susceptibility data, this command allows the user to add a string of text. When written to the .dat file, it also includes the time stamp.

**Figure 3-10:** Datafile Comment sequence command dialog.



### 3.3.3 New Datafile

It is occasionally convenient in longer, more complex sequences to be able to systematically change which datafile results are being written. The New Datafile command allows the user to specify a location (**Path**), **Name**, and **Title** for the file using the **Browse...** button. If the file does not already exist, it will be created upon execution of the sequence command. If a file with a matching name already exists at the specified path, the setting under the **File Action** pane determines what occurs. **Create New File/Version** will cause a new file to be created with a matching name and **\_00001** added to the end (running the same command subsequent times will continue creating new files, incrementing the appended number by one each time). **Append to File** will simply continue adding data to the existing file.



**Figure 3-11:** New Datafile sequence command dialog. Note: the checkbox for the option to Record Raw Data is not checked by default.

This dialog also includes the checkbox to indicate that the option should **Record Raw Data**, which is occasionally useful for diagnostic purposes.

## 3.4 File Structure

For a specified location and datafile name, an AC susceptibility measurement will always generate a corresponding .dat file which includes the AC susceptibility values, measurement parameters, and key environmental variables. If raw data is being recorded the option software will also generate a .raw file containing that information as well. The content of the columns of these two files is described briefly in the following sections.

### 3.4.1 Measurement File Data Columns

**Table 3-1:** Data columns included in ACDR Option-produced .dat files.

Field Name	Units	Description
Comment		Text string included by the Datafile Comment command
Time Stamp	minutes, relative	When displayed as 'relative', the time dates from the creation of the data file; when displayed as 'absolute', the time is measured since the epoch of January 1, 1900.
Temperature	K	Temperature reported by the DR sample stage; averaged across the duration of the measurement
Magnetic Field	Oe	Applied DC magnetic field; averaged across the duration of the measurement
AC Moment	emu <sup>4</sup>	Magnitude of AC moment signal
AC Std. Dev.	emu	Standard deviation of the population of corresponding measurements extracted from individual waveforms acquired during the total elapsed sampling time
AC Phase	degrees	Phase angle between 'AC Drive' and 'AC Suscept.' signals
AC Phase Std. Dev.	degrees	Standard deviation of the population of corresponding measurements extracted from individual waveforms acquired during the total elapsed sampling time
AC Suscept. <sup>5</sup>	emu/Oe	Quotient of 'AC Moment' and 'AC Drive'
AC Suscept. Std. Dev.	emu/Oe	Standard deviation of the population of corresponding measurements extracted from individual waveforms acquired during the total elapsed sampling time
AC X'	emu/Oe	Real (in-phase) component of the AC susceptibility
AC X''	emu/Oe	Imaginary (out-of-phase) component of the AC susceptibility
AC X' Std. Dev.	emu/Oe	Standard deviation of the population of corresponding measurements extracted from individual waveforms acquired during the total elapsed sampling time
AC X'' Std. Dev.	emu/Oe	Standard deviation of the population of corresponding measurements extracted from individual waveforms acquired during the total elapsed sampling time
AC Drive	Oe	Amplitude of the AC excitation field
AC Frequency	Hz	Frequency of the AC excitation field
Range	mV	Largest measurable voltage for a given amplification setting
Measure Count		Number of drive cycles elapsed during sampling window

<sup>4</sup> See [Section 1.2.2.2](#) for additional information about the designator 'emu'.

<sup>5</sup> See [Section 1.2.2](#) for a discussion concerning the use of the term 'susceptibility' in the context of the ACDR Option.

Field Name	Units	Description
Measure Time	seconds	Approximate time elapsed during measurement
Measure Number		In a measurement comprised of multiple amplitude and frequency combinations, this number identifies where a particular measurement falls in the list of combinations (like the one shown in <a href="#">Figure 3-8</a> ).
AC X' Raw	emu/Oe	Real component of the AC susceptibility before applying factory-calibrated corrections for issues such as the image effect and frequency roll-off of drive/signal attenuation
AC X'' Raw	emu/Oe	Imaginary component of the AC susceptibility before applying factory-calibrated corrections for issues such as the image effect and frequency roll-off of drive/signal attenuation
Coil InPhase	V	Average amplitude of the in-phase component extracted from detection coil response voltage
Coil Quadrature	V	Average amplitude of the out-of-phase component extracted from detection coil response voltage
Null Status	code	Valid states include: 0 = no nulling signal applied; 2 = result after nulling determined in calibration; 6 = result after nulling signal from .cfg file applied;
Trim Ratio		Ratio between the current amplitude energizing the excitation coil and that supplied to the trim coil for nulling
Trim Phase	degrees	Phase angle between the current energizing the excitation coil and that supplied to the trim coil for nulling
Max. Signal	mV	Maximum recorded value of the raw coil voltage signal
Block Temp.	K	Temperature of the base PPMS; represents the temperature of the ACDR coil set
Min. Temperature	K	Lowest reading from the DR sample stage occurring during the measurement
Max. Temperature	K	Highest reading from the DR sample stage occurring during the measurement
Min. Field	Oe	Lowest reading from the magnet power supply occurring during the measurement
Max. Field	Oe	Highest reading from the magnet power supply occurring during the measurement
Pressure	Torr	Pressure reported by the PPMS during the measurement
Temp Status	code	Refer to the PPMS or DynaCool manuals to interpret base system status codes

Field Name	Units	Description
Field Status	code	Refer to the PPMS or DynaCool manuals to interpret base system status codes
Chamber Status	code	Refer to the PPMS or DynaCool manuals to interpret base system status codes
Measure Status	code	Refer to the PPMS or DynaCool manuals to interpret base system status codes

### 3.4.2 Raw File Data Columns

**Table 3-2:** Data columns included in ACDR Option-produced `.raw` files.

Field Name	Units	Description
Time Stamp	minutes/seconds, relative/absolute	When displayed as 'relative', the time dates from the creation of the data file; when displayed as 'absolute', the time is measured since the epoch of January 1, 1900.
Measure No.		Counts data points sequentially in the order written to the file
Measure Type		Always indicates AC
Point Type		Always indicates 1 (single-point measurement)
Temperature	K	Temperature reported by the DR sample stage; averaged across the duration of the measurement
Field	Oe	Applied DC magnetic field; averaged across the duration of the measurement
Drive	Oe	Amplitude of the AC excitation field
Frequency	Hz	Frequency of the AC excitation field
Gain		Upper limit of amplifier used to sense induced voltage
Position	mm	Number of drive cycles elapsed during sampling window
Ch1_inPhase	V	Average amplitude of the component extracted from detection coil response voltage which is in-phase with the current in the drive coil
Ch1_quadrature	V	Average amplitude of the component extracted from detection coil response voltage which is out-of-phase with the current in the drive coil
Ch2_inPhase	V	Analogous to Ch1_inPhase but includes the calibration coil in series with the detection coils
Ch2_quadrature	V	Analogous to Ch1_quadrature but includes the calibration coil in series with the detection coils

Field Name	Units	Description
Trim ratio		Ratio between the current amplitude energizing the excitation coil and that supplied to the trim coil for nulling
Trim phase	degrees	Phase angle between the current energizing the excitation coil and that supplied to the trim coil for nulling
Null Status	code	Valid states include: 0 = no nulling signal applied; 2 = result after nulling determined in calibration; 6 = result after nulling signal from .cfg file applied;
Ch1_maxSignal	V	Highest reading of Ch1 voltage occurring during the measurement
Ch2_maxSignal	V	Highest reading of Ch2 voltage occurring during the measurement

## 4.1 Measurement Procedures

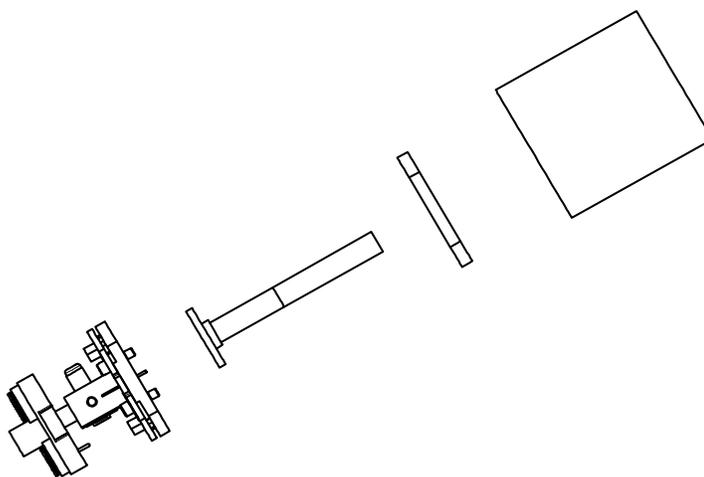
In order to achieve an accurate measure of a sample's dynamic susceptibility it is necessary to first perform the nulling procedure for the ACDR coil set. Once the nulling procedure has successfully been completed the sample can be installed and the measurement can commence.

### 4.1.1 Nulling Procedure

These directions assume the DR is warm and has been removed from the PPMS with no other measurement options installed on the sample stage.

#### 4.1.1.1 Attach the ACDR Sample Holder

Choose which sample holder stand-off will be used for the experiment, either the perpendicular or parallel configuration (see [Section 2.3](#) for more information). Thread the post through the hole in the ACDR sample mount plate and set this onto the DR sample stage. The DR sample stage is keyed with a very small pin located to one side; a corresponding hole in the mount plate ensures the threaded holes for the screws are properly oriented. Tighten the two captured screws nearly all the way, such that the standoff can still be translated to a small degree in the plane of the stage.



**Figure 4-1:** Installing the ACDR sample holder. At the bottom left the sample stage of the DR is shown; the sample holder (parallel variant shown) interfaces directly with the stage and is held in place by the mounting plate. The radiation shield shown at top right is installed last over the completed assembly after the sample holder has been centered.

### 4.1.1.2 Center the Sample Holder

Gently place the sample centering fixture onto the DR and slide it down until it stops. Examine the position of the sapphire stand-off from above and attempt to position it concentrically within the aperture of the sample centering fixture. When it is reasonably centered, tighten the screws the final few turns until snug. Remove the sample centering fixture. Install the radiation shield, being careful not to cross-thread it; if abnormal resistance is encountered, remove, clean the threads and try again.

### 4.1.1.3 Prepare System for Option Installation

If it is not already warm, bring the PPMS up to 300 K and the magnetic field down to 0 Oe. Vent the sample chamber continuously and remove any pucks or other hardware still inside the sample chamber. Be sure to remove the standard KF centering ring/O-ring since the dilution refrigerator has its own sealing surface to interface with the sample chamber flange.

Now is also a good time to flex the contact fingers of the DR and apply fresh vacuum grease to ensure adequate thermal contact, as well as to cryo-clean the gas mixture if necessary. For more information, see the Dilution Refrigerator Option User's Manual.

### 4.1.1.4 Install the ACDR Coil Set

Note the serial number written on the fiberglass base of the ACDR coil set before using the PPMS puck extraction tool to install it in the bottom of the sample chamber. Be mindful that the puck is keyed and will only 'drop' to interface with the sample chamber pins if it is in the correct orientation.

### 4.1.1.5 Install the Dilution Refrigerator

While the sample chamber is still venting, remove the DR from its cart, invert it, and insert into the sample chamber. It is typically useful to do this while standing on a short stool, given the height of a typical PPMS standing on the floor. It is also advised to have someone assist in guiding the bottom end of the probe into the sample chamber- if not aligned properly, the exposed sapphire stand-off can be damaged should it collide with the sample chamber flange on install.

Take care also when installing the DR not to apply excessive force to the top of the probe during insertion- this can bend the probe and irreparably damage the internal gas-handling system. When the probe is fully installed, vent and seal the sample space.

### 4.1.1.6 Finalize System Connections

Connect the round LEMO-style connector of the ACDR signal cable to the grey LEMO socket on the PPMS and the 15-pin D-sub connector to the ACDR Module (JM-1). Make sure the RF-filter cable for the DR temperature control is also connecting the DR to the corresponding module.

### 4.1.1.7 Run the Nulling Calibration Wizard

Activate the AC Susceptibility for DR option (**Utilities>Activate Option...**) within MultiVu. Confirm that the reported serial number in the AC DR Log window reading "**Loading coil configuration file: ACDRCOIL-xxx.CFG**" corresponds to the installed coil set, and that the option successfully initializes.

The nulling wizard is accessed via **Utilities>AC DR>Wizards>CalibrateNulling**. The wizard is fully automated and will handle all temperature control and nulling procedures. The process typically takes on the order of 10 – 15 hours but may vary depending on a particular dilution refrigerator's performance. As the wizard proceeds with the nulling procedure the results are recorded in the `\ACDR\Calibration\` folder with a unique identifying number appended. The wizard automatically displays this file and upon completion it can be used to evaluate if the nulling procedure has succeeded.

As described in [Section 1.2.3](#) it is best to perform the nulling at the same frequencies that will be used for the measurements. The default nulling frequencies can be viewed by opening the `CalibrateNulling.BAS` file which can be found in the `c:\Qdxxx\ACDR\Wizards` directory (where `xxx` is either `Dynacool` or `Ppms`). This file can be

opened and edited with any standard text editor, e.g. Notepad or the MultiVu Scripting environment (**Sequence>Advanced>Edit Macro**), as shown in **Figure 4-2**. The default nulling frequencies are highlighted and can be modified for your specific needs. Note, a simple linear interpolation of the nulling parameters is used when measuring between frequencies.

```

Dim fn As Variant
Dim Prefix As String
Dim Center As Single
Dim i As Integer
Dim RootDir As String
Dim dataArray As Variant

dataArray = Array(0)
DriveAmpl = 3.0 ' we will lower this as needed if nulling fails due to railing
D.Text("Starting Calibration at "&Format(DriveAmpl,"0.0")&"Oe")
FreqTable = Array(10,25,50,100,250,500,1000,1500,2000,2500,3000,4000,5000,5000.1,6000,7000,8000,9000,10000)

RootDir = Utils.systemDirectory + "\Data"
CfgDir = Utils.systemDirectory + "\ACDR\Calibration"

CreateDir( RootDir )
RootDir = RootDir + "\ACDR"
CreateDir( RootDir )
RootDir = RootDir + "\NullingCalibration"
CreateDir( RootDir )
RootDir = RootDir + "\ACDR"+CoilsN
CreateDir( RootDir )

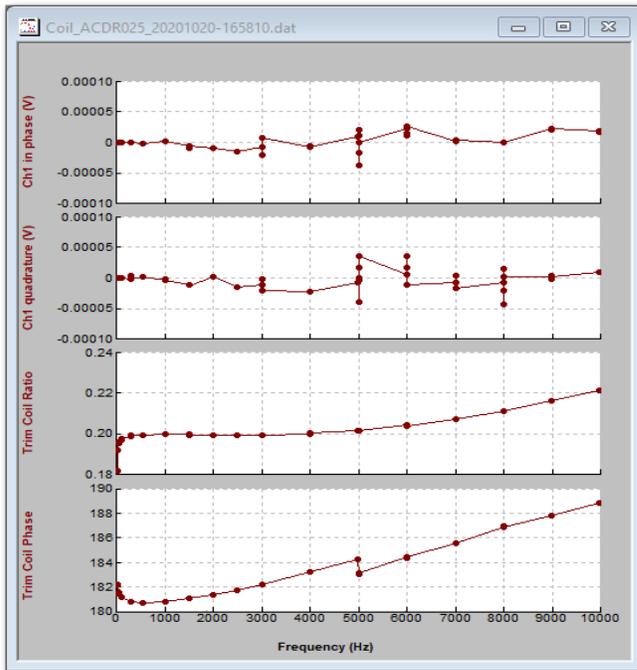
```

**Figure 4-2: CalibrateNulling.BAS** file highlighting the default frequencies used during the nulling procedure. If needed, these can be altered to match those used during measurements.

#### 4.1.1.8 Evaluating the Nulling Data

Make sure only the ‘nulled’ data is displayed by setting a filter (Right click the plot, **Data Selection**; choose **Filter**, and set the bounds of **Nulling Status** to both be ‘2’), and look for the following key features in the plot:

- a. The scatter in both **Ch1 in phase** and **Ch1 quadrature** should both be centered around zero.
- b. The maximum deviation for either **Ch1 in phase** or **Ch1 quadrature** should be  $1 \cdot 10^{-4}$  V.
- c. Both the **Trim Coil Ratio** and **Trim Coil Phase** should be smooth, continuous functions with minimal scatter, with the exception of a small step feature in the **Trim Coil Phase** at 5 kHz, which is typical and expected.



**Figure 4-3:** Example dataset showing successful detection coil nulling. The scatter in the **Ch1 in phase** and **Ch1 quadrature** plots is both centered about zero and does not extend beyond  $\pm 1 \cdot 10^{-4}$  V, while the **Trim Coil Ratio** and **Trim Coil Phase** are both smooth, continuous functions.

An example set of acceptable nulling data is provided in [Figure 4-3](#) for comparison. The table of trim coil ratio and trim coil phase dependence on frequency is automatically recorded into the `ACDRCOIL-xxx.cfg` file and configured by the wizard for subsequent sample measurements.

## 4.1.2 Sample Measurement

If the nulling wizard has finished successfully, the DR must then be completely warmed and removed. The option is now prepared for a sample measurement.

### 4.1.2.1 Sample Size and Shape Considerations

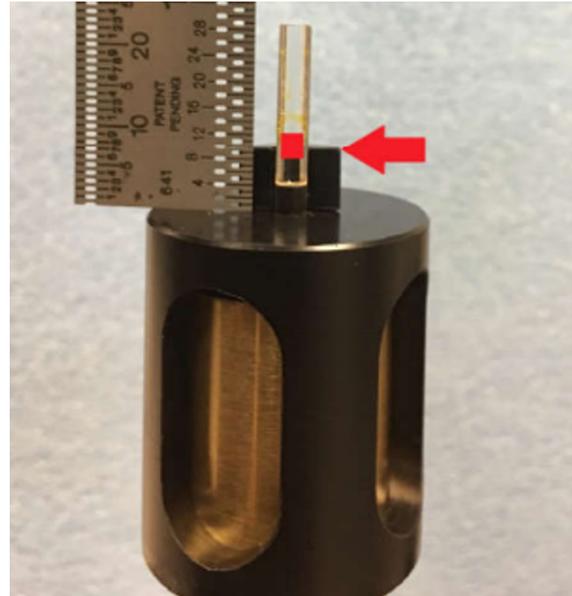
Samples should be small enough that they do not significantly hang over the edge of either style of sapphire stand-off and are not substantially longer than they are wide. Meeting these conditions ensures that the bulk of the sample material is concentrated around the center of the upper turns of the detection coil upon installation, minimizing the impact of edge effects on accuracy.

### 4.1.2.2 Adering the Sample to the Standoff

Place a small drop of dilute GE-7031 varnish (or similar removable adhesive with adequate cryogenic performance) at the location where the sample is to be mounted, then press the sample gently into the drop of varnish. If using the parallel stand-off, make sure to use the included sample height fixture along with the centering fixture as shown in [Figure 4-4](#) to make sure the sample will be at the correct height inside the coil set.

Using as little varnish as needed is recommended, to keep any signal contribution of the adhesive from significantly impacting the accuracy of the result. Give the varnish ample time to dry, as the DR operates in high-vacuum: if any trapped air is drawn out of the varnish drop before it dries, there is a risk of the resulting bubble dislodging the sample.

**Figure 4-4:** Proper sample mounting location using the sample centering fixture (4091-707) and ACDR sample height fixture (4091-621) when using the parallel sapphire rod.



### 4.1.2.3 Commence Sample Measurement

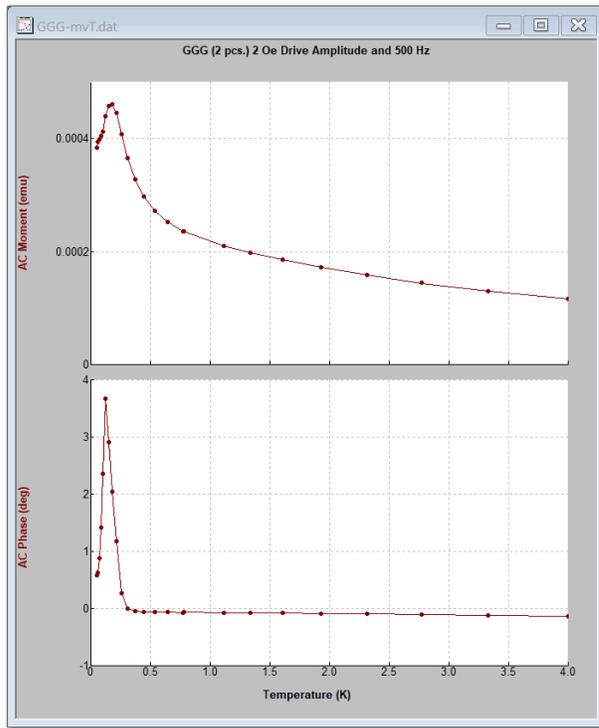
Repeat the process described in Section 4.1.1.5 to install the DR, now with the sample mounted, into the PPMS sample chamber. Once the DR has cooled down to within its operating regime (4 K – 0.05 K) the system is ready to begin measurements of the sample. For more information on measurement configuration and sequence commands see [Section 3.2](#) which describes the software interface in detail.

### 4.1.3 Example Measurement

[Figure 4-5](#) shows a measurement of two GGG (4091-609) reference chips (mounted on top of one another) using GE-7031 varnish and the parallel sapphire sample holder (4091-616-01). This particular measurement spans the entire temperature range available for the ACDR option and shows measurement data acquired using a 2 Oe AC Drive and 500 Hz AC Frequency excitation. For temperatures above 1 K the AC Moment of the GGG exhibits the classic  $1/T$  response (Curie's law) and the AC Phase is near zero, as expected of a near-ideal insulating paramagnet. Interestingly, just below 200 mK a clear peak in the AC susceptibility is observed, consistent with a highly frustrated spin configuration [4]. Note, these results are intended to simply provide an instructive measurement example of the included reference sample and are not necessarily intended to serve as a rigorous calibration standard.

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[4] [arXiv:1501.07229v1](https://arxiv.org/abs/1501.07229v1)



**Figure 4-5:** A measurement of two GGG reference chips acquired using a 2 Oe AC Drive and 500 Hz AC Frequency excitation. A clear peak in the AC susceptibility just below 200 mK is observed, consistent with a highly frustrated spin configuration.

## 4.2 Removing the ACDR Option

When the experiment is complete, the DR should be warmed to 300 K and given sufficient time to equilibrate (the probe has a substantial thermal mass, and typically needs on the order of four hours or more at 300 K to fully warm; venting and sealing the system once warmed to 300 K can expedite this). Deactivate the ACDR Option software, vent the system, then remove the DR and the ACDR coil set. Disconnect the cabling between the ACDR Module and the PPMS, as well as the DR temperature control cable. Store all loose cables and hardware in a safe place.

Purge and seal the sample chamber so it is ready for the next user. Carefully remove the sample from the stage after using a solvent to soften the GE-7031 (typically a 50:50 by volume mix of ethanol and toluene), and gently clean the remaining varnish from the holder. If this is done cautiously and the sample holder is not shifted in its mounting plate, the stand-off can be left on the DR stage and nulling does not need to be repeated for the next ACDR measurement. If the sample holder is removed, or switched to the other type, a new nulling procedure should be run.

### 5.1 Cleaning Hardware

#### 5.1.1 Sample Holders

From time to time it is a good idea to occasionally look over the sapphire stand-offs to make sure they have not accumulated excess varnish or grease in the vicinity of where the sample is placed. Wipe off any foreign material with a mild organic solvent and inspect visually to confirm the sample holders are clean. If they are pitted or chipped, contact Quantum Design to order a replacement- this marring can introduce additional background signal.

Similarly, the base of these posts along with the sample mount plate used to affix them to the DR stage should have a thin layer of N-grease for purposes of thermal conduction. If this grease is missing or has become dirty, wipe the contact surfaces gently and spread a small amount of grease into a very thin layer. Not much is needed since the surfaces will be screwed down snugly to the DR stage.

#### 5.1.2 ACDR Coil Set

Occasionally samples may fall off inside the coil set- simply invert it so shake out any loose material. The inside bore of the coil set may be cleaned with a mild organic solvent and a cotton swab; the coils are not mechanically accessible from the inner bore, so no special care must be taken while cleaning, just handle the coil set gently as usual.

## Initial Installation

### A.1 Use of This Guide

If the ACDR Option has never been used the following sections will guide the user through the process of the initial setup. These steps are only intended to describe the installation of the ACDR Option for the first time. Part or all of this procedure may have been complete already if the option shipped with the base system from Quantum Design. In this case, or once the initial user installation has been completed, the option is simply activated when needed, as described later in [Section 4.1](#).

#### A.1.1 Prerequisites

The ACDR Option is only compatible with platforms capable of supporting the Dilution Refrigerator Option, which are the DynaCool and PPMS (without EverCool II). Beyond a compatible base system, a few extra components are necessary for the successful installation and operation of the ACDR Option:

- Dilution Refrigerator Option
- Model 1000 Modular Control System (PPMS only)
- Cryopump High-Vacuum Option (PPMS only)
- MultiVu software

If some of these items still need to be installed, refer to their corresponding manuals and complete the installations. If any of these items are missing or not working properly, contact [service@qdusa.com](mailto:service@qdusa.com) to resolve the issue before proceeding with the ACDR installation.

#### A.1.2 Option Software Installation

Before continuing, it is also a good idea to ensure that the base installation of MultiVu is up-to-date with the current version. This software can be found under either the DynaCool or PPMS sections of the 'Software Upgrades' section of the Quantum Design website.

Once the MultiVu installation is current, the ACDR Option software can also be downloaded from the same page. With MultiVu closed, run the installation wizard for the ACDR. When the installation is complete, browse to the root folder of the instrument (usually `C:\QdPpms` or `C:\QdDynaCool`) and locate `\ACDR\Calibration\`. A temporary configuration file (`ACDRCOIL-xxx.cfg`) resides there; copy the correct configuration file that shipped with the ACDR Option into this same directory, making note of the serial number appearing in the `xxx` position of the file name.

When these steps are complete, proceed to install the ACDR Module.

#### A.1.3 Installing the ACDR CAN Module

First verify that all of the items on the Key Components list (near the start of this manual) were included with the ACDR Option and that they appear undamaged. If everything is in order, continue on to installing the CAN Module.

- a. Ensure that all options have been deactivated in MultiVu.
- b. Power down the CAN rack (DynaCool) or the Model 1000 (PPMS).



**Warning:** Never install or remove a CAN module while the module bay is still powered. This can permanently damage the electronics. Never install or remove a CAN module while the module bay is still powered. This can permanently damage the electronics. Never install or remove a CAN module while the module bay is still powered. This can permanently damage the electronics.



**DANGER:** Do not put a piece of flatware into a live electrical socket - you could hurt yourself!

- c. Identify an empty module bay and remove the cover plate; if all bays are full, remove a module that will not be need.
- d. Insert the ACDR module into the empty bay. (PPMS Note: the module is keyed for the 'high-power' style bays and will only fit in the rear three positions of the Model 1000.)
- e. Re-power the modules.

#### A.1.4 Confirm Option Activation

With the software and electronics installed and the correct configuration file in place, the system is ready to be activated for the first time:

- a. Open the **Utilities>Activate Option** menu in MultiVu to bring up the Option Manager
- b. Select the **AC Susceptibility for DR** item and click **Activate**.
- c. Check the AC DR Log window; was the correct `.cfg` file loaded? If so, no additional action needed; the option was successfully installed, proceed to 6.
- d. If not, choose the **Configure** tab of the AC DR window and press **Change Coil Configuration**
- e. Type in the serial number of the `.cfg` file copied to `\ACDR\Calibration\` in [Section A.1.2](#) and press **Next**, then **Finish** when the configuration is loaded.
- f. The ACDR Option is now properly configured. The option software can be deactivated if a measurement will not be conducted right away. If the user is ready for a measurement, proceed to Chapters 3 and 4.

## A.2 Subsequent Installations

If the steps comprising the initial installation are already complete, refer to [Section 4.1](#) for details outlining a typical activation of the ACDR Option once installed.



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