



AC Susceptibility (AC Measurement Option-ACMSII)

<http://education.qdusa.com/experiments.html>

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The goal of this Educational Module is to guide the student through the basic theory behind, and the steps required for, measuring the magnetic properties of solids using the ACMS II option for the Quantum Design VersaLab and PPMS.

Introduction:

This section will provide a brief introduction to the differences between DC and AC measurements and how AC measurements are performed.

Differences between DC and AC susceptibility

The intrinsic quantity magnetization (M) of a substance is related to the applied magnetic field (H) by the well-known relation:

$$M = \chi H \quad (\text{Eqn. 1})$$

where χ is defined as the *magnetic susceptibility*.

In order to determine the DC susceptibility χ_{DC} of a sample, an external magnetic field (H) is first applied to magnetize the material. The value of M can then be determined using a variety of techniques including, faraday balance, vibrating sample magnetometry (VSM), SQUID, etc. and the equation above can simply be applied.

In fact, measurements of the DC moment, analogous to the dedicated VSM option, can also be carried out with the ACMS II. A magnetic field is applied to magnetize the sample, which is positioned between the two detection coils

shown in red, Fig. 1. The sample is then vibrated sinusoidally around the center position. The physical oscillations of the sample induce a time-varying flux that is detected in both pickup coils. The resulting AC voltage signal is proportional to the DC moment, frequency, and amplitude of vibration, and therefore the VSM can be calibrated to convert this induced voltage from the pickup coils to a DC moment value.

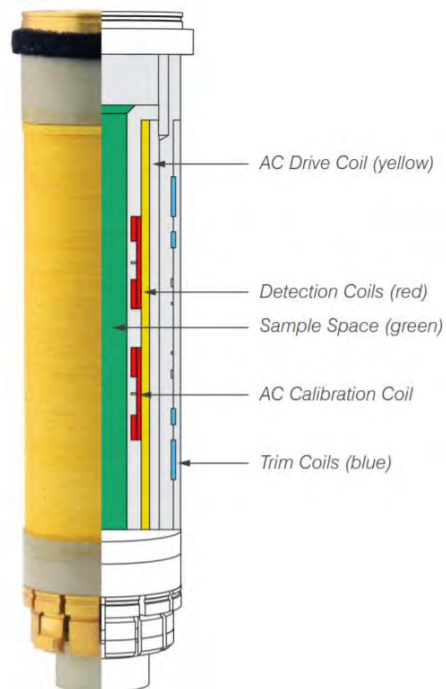


Figure 1: Schematic view of the ACMS II coil set internal components highlighting the various individual constituent coils.

The ACMS II Option is specifically designed for measuring the *dynamic, or AC, susceptibility* of a material as a function of the AC magnetic field magnitude/frequency and/or the environmental parameters of temperature and/or DC magnetic field.

Origins of the AC susceptibility signal

Consider the ACMS II coil set's detection coil (Fig. 1): a pair of counter-wound turns (each numbering n) of a single metallic wire centered concentrically in the 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 is installed, it resides at the center of one of these sets of turns; in this way, one set of turns see 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 AC excitation field, generated by the AC drive coil shown in yellow in Fig. 1.

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 $\mathbf{m}(H)$ curve that would be measured in a DC experiment, only now the applied field is the time-dependent $H = H_{AC} \cos(\omega t)$. For this limiting case, what is the resulting voltage induced in the detection coil? Starting from Faraday's Law:

$$\mathcal{E} = -\frac{d\Phi}{dt} \quad (\text{Eqn. 2})$$

...where the magnetic flux Φ can be understood as the surface integral (across the area of both sets 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:

$$\Phi = n[\oint \mathbf{B}(\mathbf{r}, \mathbf{m}) \cdot d\mathbf{A}_1 - \oint \mathbf{B}(\mathbf{r}, \mathbf{m}) \cdot d\mathbf{A}_2] \quad (\text{Eqn. 3})$$

A few points to note about the above expression: First, the two surface integrals 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 $\hat{\mathbf{m}}$ of the moment vector \mathbf{m} , but also the position in space \mathbf{r} for the various points across the surface bounded by the turns of wire. It can be shown that the magnitude of the magnetic field from a dipole is actually linear in the size of the moment vector, m , so we can re-write:

$$\Phi = m \cdot \{n[\oint \mathbf{B}(\mathbf{r}, \hat{\mathbf{m}}) \cdot d\mathbf{A}_1 - \oint \mathbf{B}(\mathbf{r}, \hat{\mathbf{m}}) \cdot d\mathbf{A}_2]\} \quad (\text{Eqn. 4})$$

...where $\mathbf{B} = \mathbf{B}(\mathbf{r}, \mathbf{m})/m$, 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 \mathcal{C} . This substantially simplifies the expression for induced voltage:

$$\mathcal{E} = -\frac{d}{dt}[m\mathcal{C}] = -\mathcal{C}\frac{dm}{dt} \quad (\text{Eqn. 5})$$

Recall that the sample moment is a function of time by way of its dependence on the magnetic field H , so the total derivative is evaluated as:

$$\mathcal{E} = -C \frac{\partial m}{\partial H} \cdot \frac{dH}{dt} \quad (\text{Eqn. 6})$$

$$\mathcal{E} = C\omega \frac{\partial m}{\partial H} \cdot H_{AC} \sin(\omega t) \quad (\text{Eqn. 7})$$

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 ω 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 and contrasts with a DC experiment where m itself is measured. One advantage of this technique is now immediately evident: since the AC measurement is sensitive not to the absolute 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.

Extending our discussion to the response at higher frequencies more typical of most AC measurements (100's or 1000's of Hz or more), *the AC moment of the sample does not generally match the slope of the DC moment curve* due to dynamic and dissipative effects in the sample. For this reason, the AC susceptibility is often known as the 'dynamic susceptibility.' In this higher frequency case, the moment 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 shift ϕ (relative to the drive signal).

The two representations are related by the following equalities:

$$\begin{aligned} X' &= X \cos \phi & X'' &= X \sin \phi \\ X &= \sqrt{X'^2 + X''^2} & \phi &= \text{atan2}(X'', X') \end{aligned} \quad (\text{Eqns. 8})$$

...where $\text{atan2}()$ is the two-argument atan2 function.¹

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

The relationship between (X', X'') and (X, ϕ) can also be represented in the complex plane, as shown below in Fig. 2.

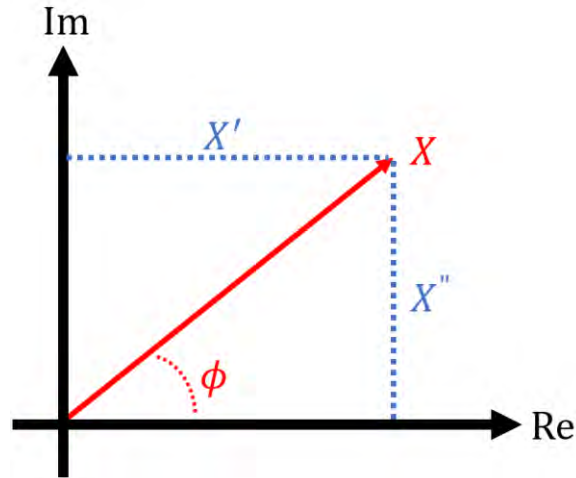


Figure 2: Graphical representation of the relation between (X', X'') and (X, ϕ) .

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 (*i.e.*, $\phi \approx 0^\circ$). For samples where some loss/dissipation 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.

Measured Quantities

A sample's true dynamic (AC) susceptibility has real (in-phase) and imaginary (out-of-phase) components to its response, indicated by χ' and χ'' respectively (this notation, lacking a subscript, typically implies the volume susceptibility). It is important to note that the ACMS II option does not report this volume susceptibility strictly speaking, but instead the real and imaginary components of the slope $\frac{\partial m}{\partial H}$. This quantity is represented in the ACMS II software by the Latin character X . For an AC measurement, the volume susceptibility χ and reported quantity X are related by:

$$\begin{aligned}\chi' &= \text{Re} \left[\frac{\partial M}{\partial H} \right] = \frac{1}{V} \cdot \text{Re} \left[\frac{\partial m}{\partial H} \right] = \frac{1}{V} \cdot X' \\ \chi'' &= \text{Im} \left[\frac{\partial M}{\partial H} \right] = \frac{1}{V} \cdot \text{Im} \left[\frac{\partial m}{\partial H} \right] = \frac{1}{V} \cdot X''\end{aligned}\quad (\text{Eqns. 9})$$

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

$$M = m/V \quad (\text{Eqn. 10})$$

Since a precise measure of the volume is often impractical, often a more convenient intrinsic quantity used is the mass susceptibility χ'_ρ :

$$\chi'_\rho = \frac{1}{\rho} \cdot \chi' = \frac{1}{b} \cdot X' \quad (\text{Eqn. 11})$$

...where the sample density is ρ and its mass is b , and the analogous form is implied for χ''_ρ .

This is mentioned here to stress the relation between the extrinsic quantity reported by the ACMS II Option (X'), and the intrinsic quantities most often of interest for purposes of comparison or use in further calculations (χ' , χ''_ρ). Outside of this discussion, the ACMS II manual and this Education Module 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 multiplicative scaling by volume, mass, or number of moles will still generate the corresponding intrinsic values.

Reported Units

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

Table 1: Reported Units of Select Properties.

Quantity	Units	Description
X, X', X''	emu/Oe	Susceptibility
ϕ	degrees	Phase angle
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.

Student Learning Outcomes:

- Sample mounting and sequence writing using the ACMS II option.
- Identifying signatures of magnetic phase transitions.
- Practice determining the temperature of phase transitions, by inspecting the as-measured data and post-processing.
- Estimate effective and saturation moments.

AC Susceptibility Examples:

AC Susceptibility of YBCO:

The top and bottom panels of Fig. 3 display the temperature dependence of X' and X'' for a thin film of superconducting $\text{YBa}_2\text{Cu}_3\text{O}_7$, respectively. The superconducting transition temperature (T_C) can be clearly seen near 85 K. The negative plateau for $T < T_C$ in the X' indicates a strong diamagnetic response, as expected in the superconducting phase. The peak in X'' centered near T_C reflects the absorption of energy as the sample transitions between the normal to the superconducting states.

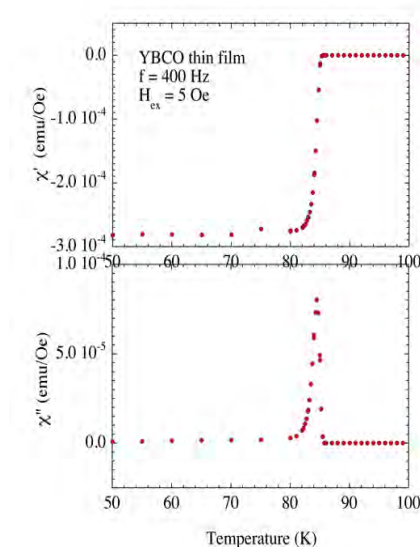


Figure 3: X' and X'' vs temperature for a thin film of $\text{YBa}_2\text{Cu}_3\text{O}_7$

AC and DC Susceptibility of Dysprosium

The temperature dependence of χ , χ' , and χ'' for a small fragment of polycrystalline dysprosium (mass = 11.5 mg) are shown in Fig. 4. Note that the out-of-phase χ'' signal is very small, suggesting that the magnetic moments can flip without much impediment at the frequency of this measurement (400 Hz). Therefore, the values of χ and χ' overlap to a large extent. The onset of antiferromagnetic (AFM) order at $T_N \approx 178.5$ K can be clearly observed in this plot. However, the determination of T_C (≈ 85 K) typically requires a more thorough analysis and cross-checking with other measurements, e.g. neutron diffraction, specific heat, etc.

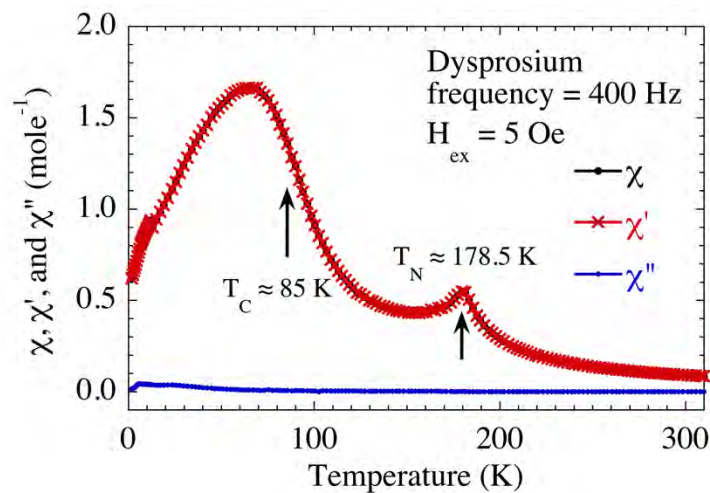


Figure 4: Temperature dependence of χ (black), χ' (red) and χ'' (blue).

The DC magnetic susceptibility of the same Dy sample measured with the VSM capabilities of the ACMS II option is shown in Fig. 5. Since $m(H)$ can depart from linearity even at low magnetic fields, and DC measurements of the susceptibility require a moderate field to magnetize the samples, most authors prefer to refer to the DC magnetic susceptibility as M/H .

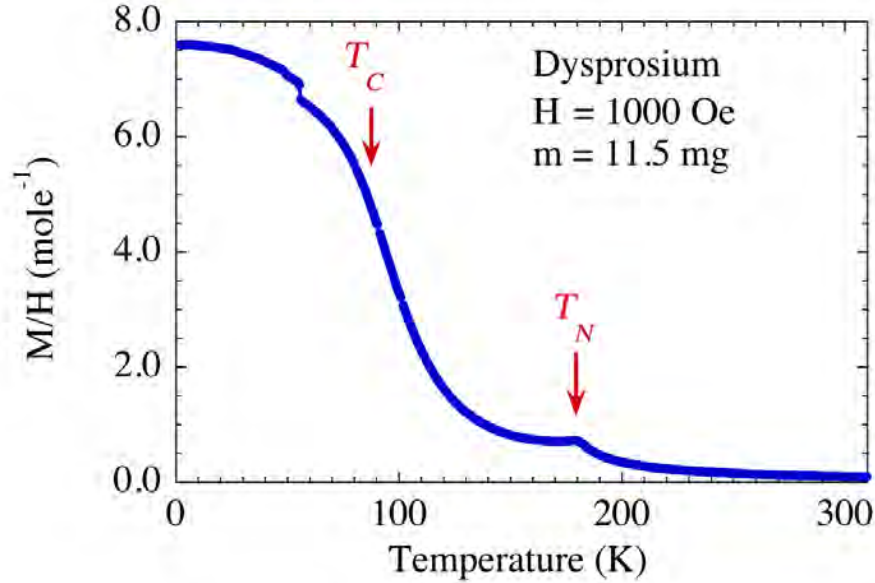


Figure 5: $(M/H)_{DC}$ vs. temperature for the same Dy sample using the VSM mode of the ACMS II. The values of T_N and T_C are indicated by arrows. The step-like feature near 56 K is an artifact, due probably to a slight repositioning of the sample inside the sample holder during the measurement.

At temperatures sufficiently above the ordering temperature, most materials with magnetic ions follow the “Curie law”, *i.e.*, for a fixed magnetic field, the magnetization is inversely proportional to the temperature. The susceptibility can be written as [4]

$$\frac{M}{H} \approx \frac{NJ(J+1)g^2\mu_B^2}{3k_B T} = \frac{Np^2\mu_B^2}{3k_B T} = \frac{C}{T} \quad (\text{Eqn. 12})$$

where M is the magnetization (in molar units), H is the magnetic field (CGS), N is the number of magnetic ions, J is the total angular momentum quantum number, g is the spectroscopic splitting factor, μ_B is the Bohr magneton, k_B is the Boltzmann constant, and p is the effective number of Bohr magnetons. To a reasonable approximation the value of p can be determined from the slope of the $(M/H)^{-1}$ vs T above the ordering temperature.

$$p \approx \sqrt{\frac{8}{\text{slope}}} \quad (\text{Eqn. 13})$$

The AC and DC susceptibility data of Figs. 4 and 5, respectively, can be used to estimate the effective moment of dysprosium. These data are shown in Fig. 6, and the estimated values of μ_{eff} obtained from the slopes of χ^{-1} vs T were 10.20

and $10.52 \mu_B$ for the AC and DC data, respectively. These values are fairly close to the theoretical value of $10.63 \mu_B$ obtained from $g[J(J+1)]^{1/2}$.

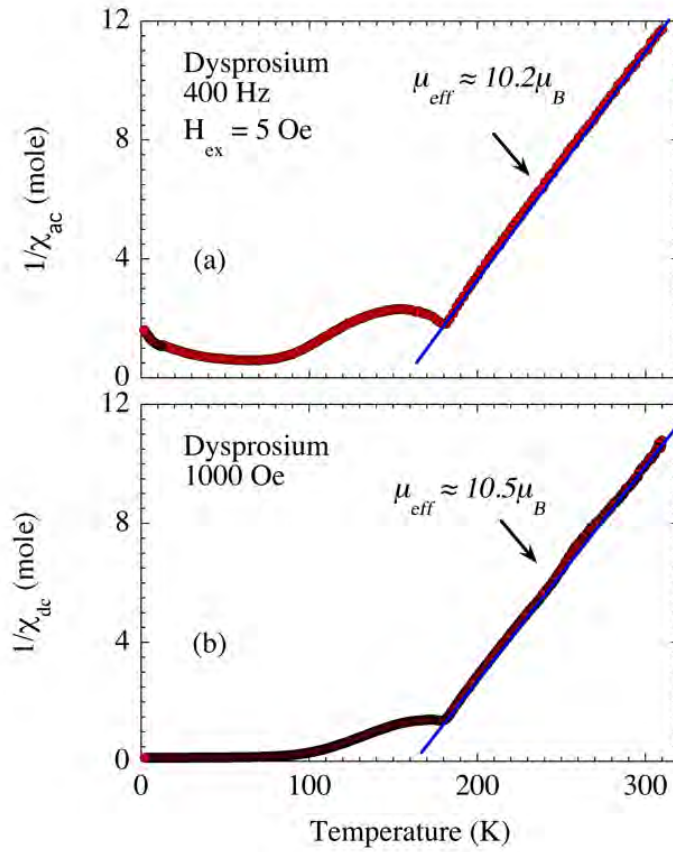


Figure 6: (a) $1/\chi'$ and (b) $1/\chi_{dc}$ vs temperature for Dy metal. The effective moment is estimated from the slopes of χ^{-1} vs T.

Safety Information:

Before attempting to perform measurements with the ACMS II, please read the entire contents of: this Educational Module, the Versalab, PPMS, or Dynacool manual, and the ACMS II manual. Observe all instruction, warnings, and cautions. These are provided to help the operators to use the equipment safely and properly, perform the experiments, and best achieve the student learning outcomes.

Quantum Design Inc. disclaims any liability for damage of the system or injury resulting from misuse, improper operation of the system, and the information contained in the Educational Module.

These safety warnings apply to this Educational Module. Read them carefully and clarify any questions that you may have with your instructor before starting the work.

WARNING!

Always use Personal Protective Equipment (PPE) during every step of sample preparation. Failure to do so might cause bodily harm.

Three circular blue icons with white symbols. From left to right: a pair of gloves, a person wearing safety glasses, and a person wearing a face shield.

Sample Mounting:

Before mounting the sample, it is recommended to first record the sample mass.

It is a good lab practice to wear nitrile gloves to handle samples and sample holders. The ACMS II accessory box has a number of resources for mounting samples, as shown in Fig. 2. Using the quartz paddle or a clear straw, the sample needs to be positioned about 25 mm from the bottom of the mounting station, as shown in Fig. 3. Using the paddle, the sample can be glued to the flat surface of the quartz using e.g. GE-7031 varnish, superglue, Duco cements, or common rubber cement. An alternative, as shown in Fig. 7, is to place the sample in a gelatin capsule, secure it by pushing in a small amount of cotton or wipe paper, and sliding the gelatin capsule inside a section of clear straw. The gelatin capsule can be secured inside the straw by small sections of folded straws on both ends. It is important that the sample doesn't move during the experiment. A potential shortcoming of using the straws is that they are more difficult to align with the rods and may therefore rub against the inside wall of the coilset, which may generate noisy erratic data, especially when vibrating the sample continuously during a DC moment measurement.

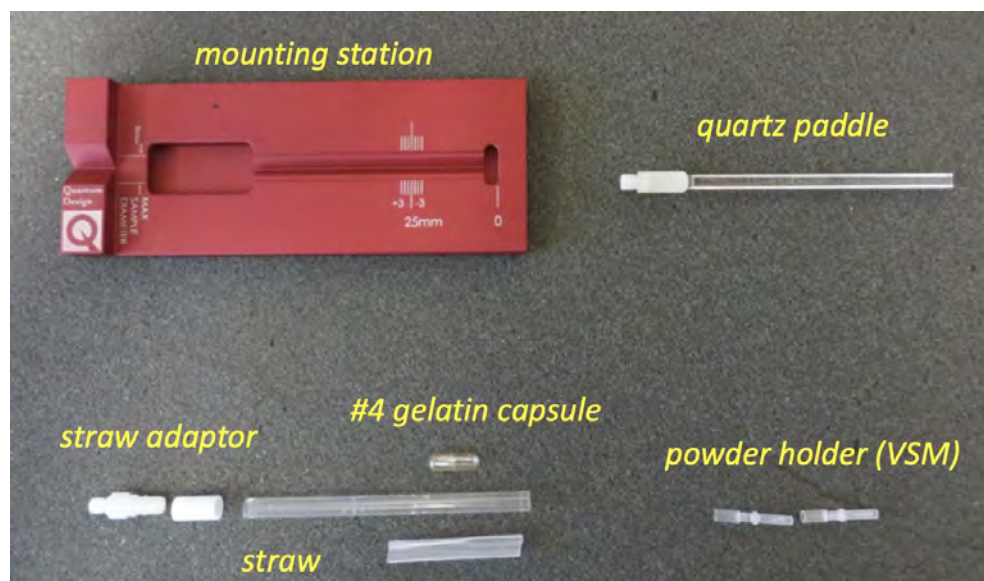


Figure 7: Mounting station, quartz paddle, straw adaptor, and powder holder for VSM measurements. Gelatin capsules are helpful when mounting samples inside of a straw.

Most important for AC susceptibility measurements is to use electrically insulating sample holders. The AC magnetic fields utilized during the measurement will generate eddy currents in a conductive sample holder, e.g. the brass half-tube

used for the VSM. These eddy currents will present an additional background and loss mechanism that will generally adversely affect the measurement accuracy. An example of sample mounting using a straw is shown in Fig. 8.



Figure 8: Sample mounting using a #4 gelatin capsule, clear straw, a straw adaptor, and wedging sections of cut straw to hold the sample in place. The sample is wrapped in a small portion of Kimwipe.

Activating the ACMS II Option:

Before loading the sample in the cryostat, one needs to install the ACMS II hardware, and activate the ACMS II option. First bring the cryostat to 300 K, vent, install the coilset (using the puck insertion/extraction tool), install the guide tube, and place the linear transport motor at the top of the cryostat. Be extra careful with the latter step; the motor head is heavy, houses a very delicate core, and can get off-balance easily. The bottom of the motor head mates with top of the cryostat using the O-ring from the guide tube and KF-40 clamp. Connect the appropriate cables between the Motor Module and the Motor and ACMS II module and grey LEMO connection.

After connecting the cables, the ACMS II option needs to be activated in MultiVu using the Option Manager, as indicated in Fig. 9.

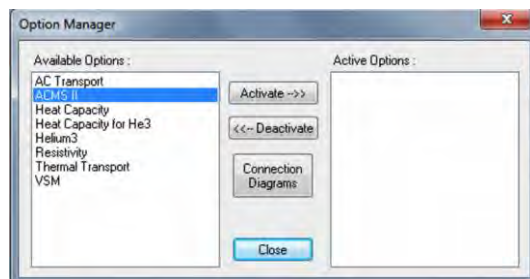


Figure 9: Activation of the ACMS II option using the Option Manager.

Monitor the log screen, Fig. 10, for confirmation that the option was installed properly, the motor limits are error-free, and the thermometer has been initialized properly.

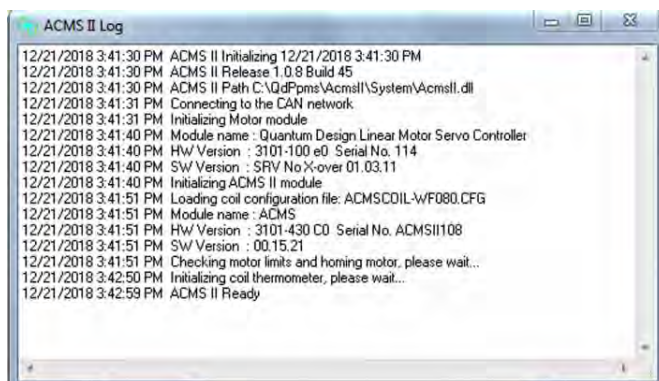


Figure 10: Activation log screen for the ACMS II.

The next step is to install the sample. Click on the “Install” tab of the *ACMS II Install/Remove Sample Wizard* dialog and click on the “Open Chamber” button shown in Fig. 11.

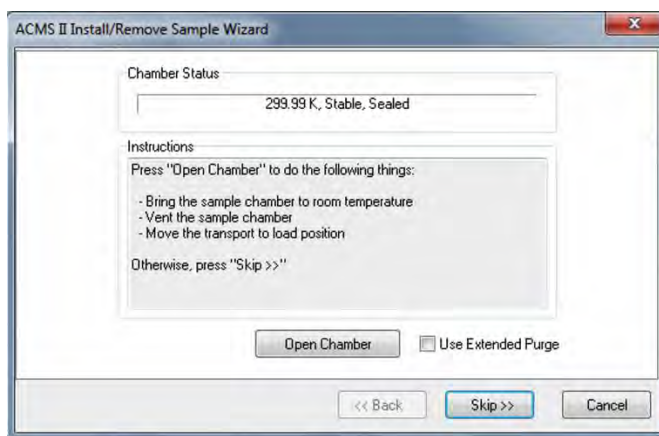


Figure 11: Install/Remove Sample Wizard.

This will ensure the sample chamber is at room temperature, vent the sample chamber with helium gas, and move the motor to the “load” position, where the motor head is ready to receive the sample rod. Lower the rod carefully until the magnet lock at the top of the sample rod “snaps” into place and put the cap on top of the motor assembly. Note, it is a good idea to check on the cleanliness status of the O-ring as you perform this step.

The next step is to enter the sample information and create an output data file, as shown in Fig. 12.

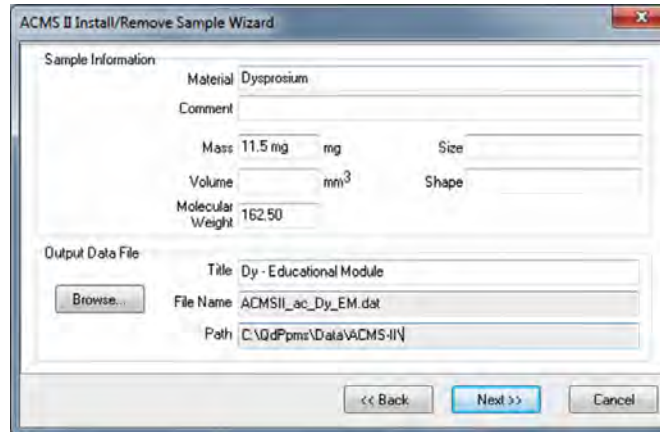


Figure 12: Creating the output file.

The wizard will then prompt you to center the sample. The dialog for centering the sample for using the AC response is shown in Fig. 13. The measured sample center position should be near 25 mm. If it is not, investigate the sample holder, either the sample has moved, or there is an impurity on the sample holder that is more magnetic than the sample of interest.

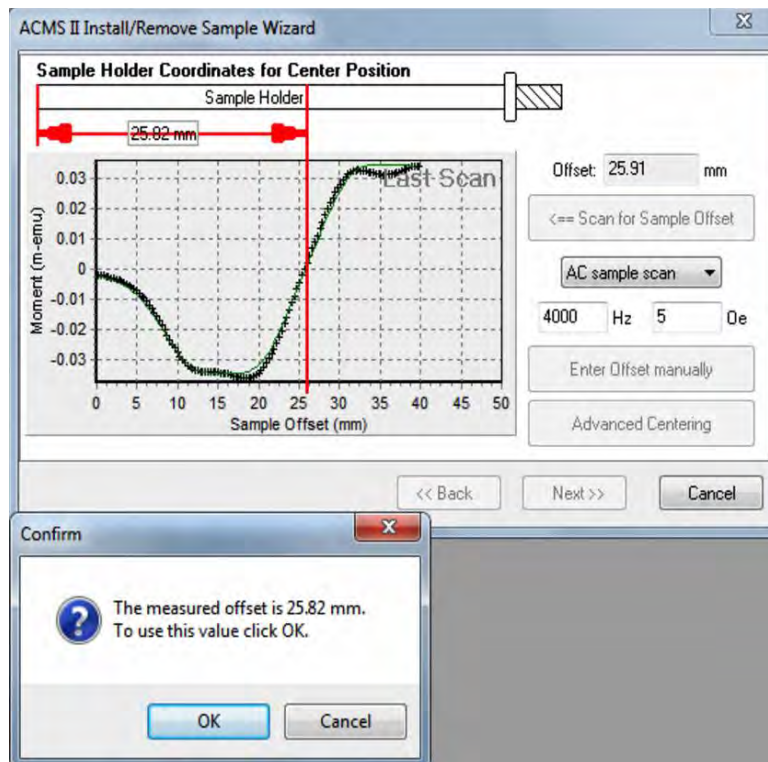


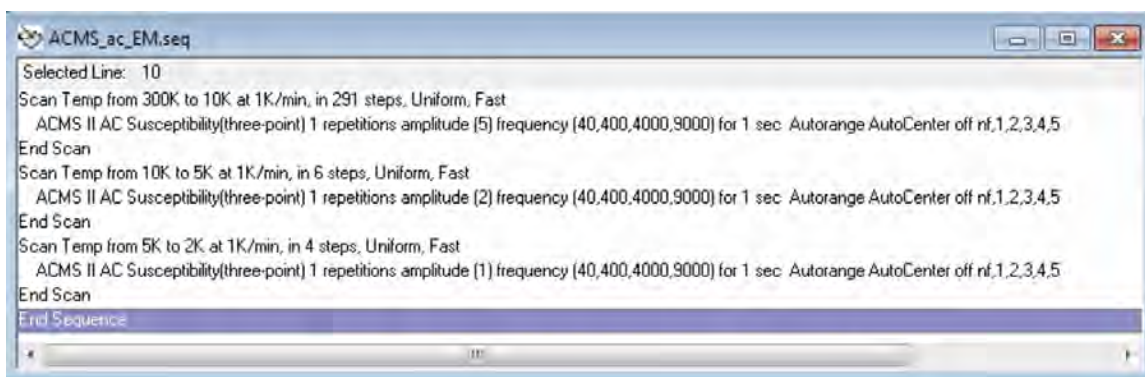
Figure 13: Sample centering using the AC sample scan.

With the sample loaded, centered, touch down done, and chamber ready, you are now ready to write a sequence to collect data.

Writing a data acquisition sequence:

The ACMS II allows for both AC and DC measurements a function of temperature and magnetic field. In either case you can collect data while sweeping the temperature or field, or scanning, *i.e.*, stopping at each temperature/field, and collecting the data. While sweeping can yield data at a faster rate, the data may not be as accurate and precise as scanning. The ACMS II option allows for excitation fields between 0.05 and 15 Oe with a frequency of 10 Hz -10,000 Hz. Note that at temperatures below 10 K, self-heating due to high frequencies and excitation fields interfere with your measurements, and one should plan accordingly, see the ACMS II manual (Table 4.2) for details. In order to minimize the errors, the default data acquisition mode is set to 3-point measurements. It is recommended measure at stable (not sweeping) temperature control points. The default vibration frequency and amplitude for the DC measurements are 40 Hz and 2 mm, respectively.

Example sequences are shown in Figs. 9 and 10 for AC and DC measurements, respectively. It is usually recommended to adjust the sequence to collect more data points near any phase transitions. For the AC sequence of Fig. 9, the temperature is first scanned from 300 to 10 K in steps of 1 K, and data is collected with an AC drive field amplitude of 5 Oe and for four excitation frequencies. Note, that for temperatures below 10 K, the excitation field is decreased in order to avoid self-heating at higher frequencies (see Table 4.2 of the ACMS II manual).



```
ACMS_ac_EM.seq
Selected Line: 10
Scan Temp from 300K to 10K at 1K/min, in 291 steps, Uniform, Fast
  ACMS II AC Susceptibility(three-point) 1 repetitions amplitude (5) frequency (40,400,4000,9000) for 1 sec. Autorange AutoCenter off nf,1,2,3,4,5
End Scan
Scan Temp from 10K to 5K at 1K/min, in 6 steps, Uniform, Fast
  ACMS II AC Susceptibility(three-point) 1 repetitions amplitude (2) frequency (40,400,4000,9000) for 1 sec. Autorange AutoCenter off nf,1,2,3,4,5
End Scan
Scan Temp from 5K to 2K at 1K/min, in 4 steps, Uniform, Fast
  ACMS II AC Susceptibility(three-point) 1 repetitions amplitude (1) frequency (40,400,4000,9000) for 1 sec. Autorange AutoCenter off nf,1,2,3,4,5
End Scan
End Sequence
```

Figure 14: Example AC Sequence.

Fig. 15 shows a simple DC moment versus temperature measurement sequence. The DC moment is measured in a 1000 Oe field from 300 to 2 K in 1 K steps.

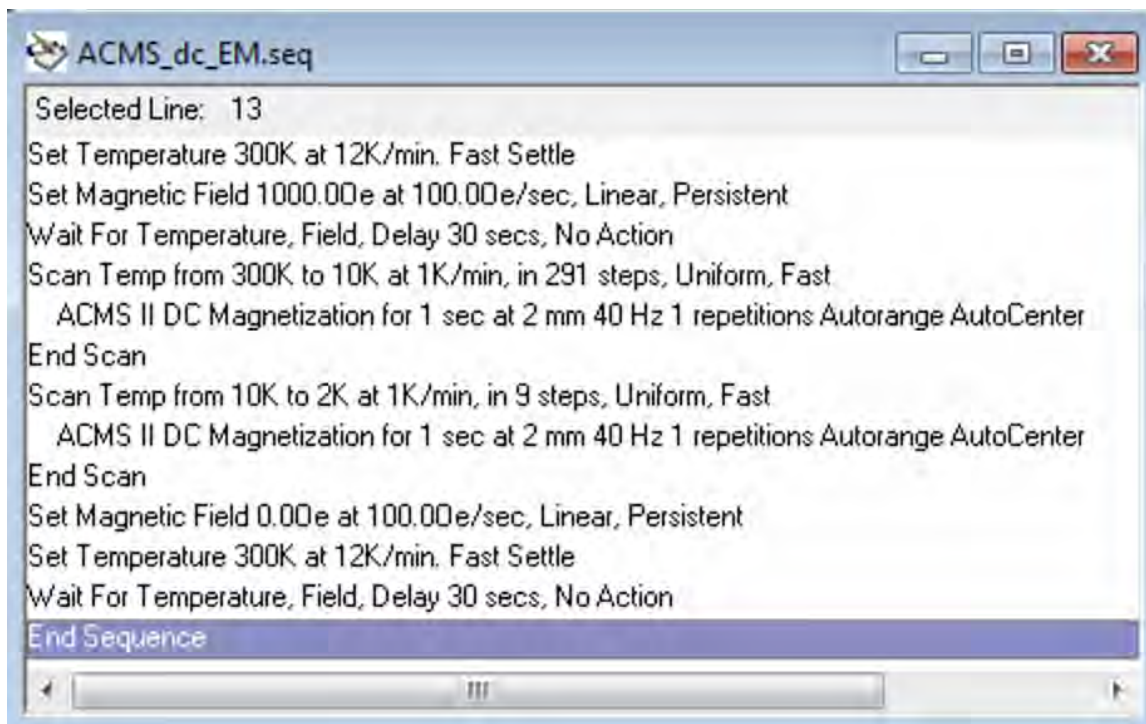


Figure 15: Example DC Sequence.

Proposed studies:

- (1) Measure the AC susceptibility as a function temperature for a variety of superconductors, e.g. $\text{YBa}_2\text{Cu}_3\text{O}_7$, Nb, Pb, etc. Refer to the [periodic table from Quantum Design](#) as a guide.
- (2) If available, measure the AC susceptibility of $\text{HoFe}_{0.5}\text{Cr}_{0.5}\text{O}_3$. Plot χ^{-1} vs T to estimate the effective moment and compare to the theoretical predictions.
- (3) Measure the AC susceptibility as a function of temperature of a heavy rare earth element, e.g. Dy, to find phase transitions. Refer to the [periodic table from Quantum Design](#) as a guide.

Discussion:

- (1) Explain the primary differences between DC and AC susceptibility.
- (2) Explain the differences between the 1-, 3-, and 5- point measurement routines used by the ACMS II option. When should each be used?
- (3) What do **periodic "touch downs"** do during the execution of a sequence. Why are they important? When should they be used?
- (4) What should one expect a difference between field-cooled and zero-field cooled experiments?
- (5) When measuring a superconducting phase transition, what physical mechanism does a peak in X'' correspond to?