

IMSERC EMX_{plus} CW X-band Manual

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INTRODUCTION

Electron Paramagnetic Resonance (EPR), is a powerful spectroscopic technique used to study materials containing unpaired electrons. The Bruker EMXplus X-band CW-EPR spectrometer is equipped with a dielectric resonator and an EMXplus standard resonator for measuring electron resonance signals in the 9.2–9.6 GHz frequency range. The Bruker VTU 3 and ESR900 enable variable-temperature experiments from 4 K to 373 K.

SAFETY

All users of IMSERC must review the general safety policies at <http://imserc.northwestern.edu/about-policies.html>. Plus, users should know the points below:

- Users with pacemaker are not allowed
- Emergency Exits
- Location of shower and eyewash
- No food and drink
- Dressing code

DATA MANAGEMENT

The personal data folder is created during training. The data were under the user's personal folder, which must be located under the supervisor's group folder.

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EPR data on this instrument are copied on 'imsercdata.northwestern.edu' under 'data2/pcm/EMX' within a few seconds. Please follow instructions at <http://imserc.northwestern.edu/about-general-faq.html#data> for details about data access.

SOFTWARE

The EPR experiment on this instrument is performed with Bruker EMXplus.

MATERIALS & METHODS

The default measurement mode of CW X-band with the loop resonator. If user want to run an experiment in a different resonator, such as dielectric resonator, they will contact the staff: Dr. Jinlei Cui. The staff will also need to tune back to the default resonator after the experiment. (these will be charged via staff time)

The default working condition of INSTRUMENT-NAME is as follows:

1. Computer screen is by default deactivated. Users must start the reservation through NUCore in order to be able to turn on the computer screen.
2. Leave the acquisition software open when user complete with the measurement.

EXPERIMENT SETUP

STEP 1. SAMPLE PREPARATION

Sample Tubes:

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a) Standard EPR tube size = 4 mm OD, however, sizes do vary (2mm, 3mm, 5mm, 10mm) and can be accommodated by the instrument. Or capillary tube.

b) EPR tubes are made out of quartz whereas NMR tubes are made out of Pyrex, which contains some iron that may or may not, depending on the sample, contribute to the EPR signal.

Tubes can be purchased by various vendors. A couple of options are,

Wilmad Glass <http://www.wilmad-labglass.com/>

Norell <http://www.nmrtubes.com/index.php>

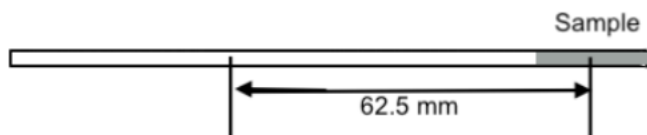
Aqueous Samples (also true for highly polar and conducting samples): The water in aqueous samples absorbs the electric part of the microwaves and begins to heat (like your microwave at home), and therefore, the sample size needs to be limited, specifically the surface area of the sample size. Current, IMSERC use capillary tubes to adsorb the liquid sample and wax to seal each open side.

Powder samples: one open side of capillary tube was sealed firstly, and power was packed into the tube.

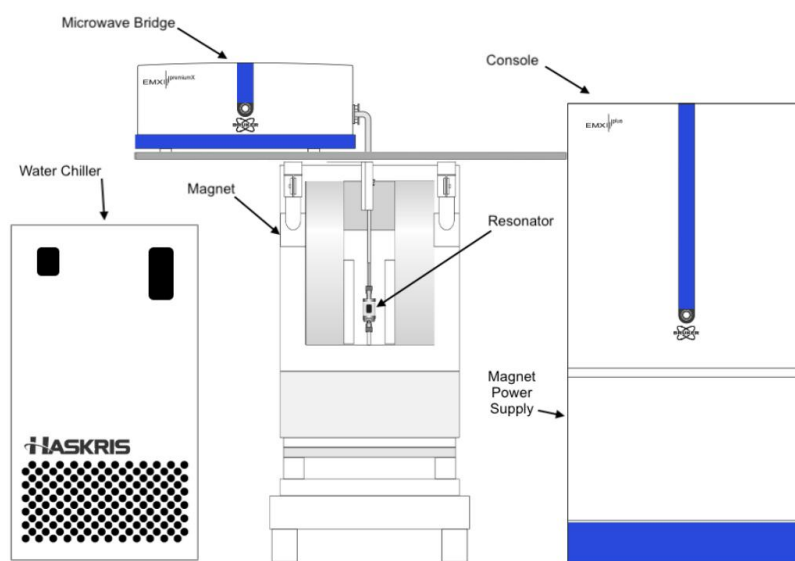
b) The samples is usually <30 mm height. **When the VTU3 setup is used, user needs to use calibrator to measure the position of 84 mm from the center of the sample and labels it with mark pen. When the ESR900 is used, user needs to use calibrator to measure the position of 162 mm from the bottom of the tube.**

However, when the VT set is not used, the length is labeled at 62.5 mm.

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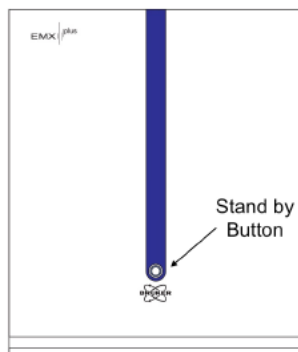
STEP 2. TURN ON THE SPECTROMETER AND OPEN THE SOFTWARE



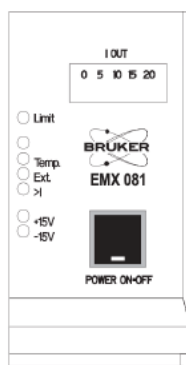
1) Turn on the following (if not already on):

Console (if it is on, then skip)

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Power Supply of the magnet

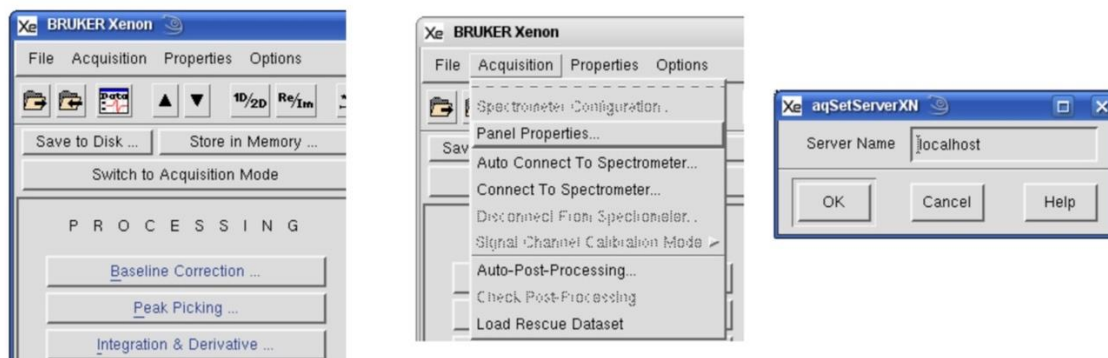


2) turn on the reservation on Nucore. Open the Acquisition software “Xenon” located on the desktop.



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3) from the “Acquisition Menu” chose “ connect to spectrometer”, then click “ok”.

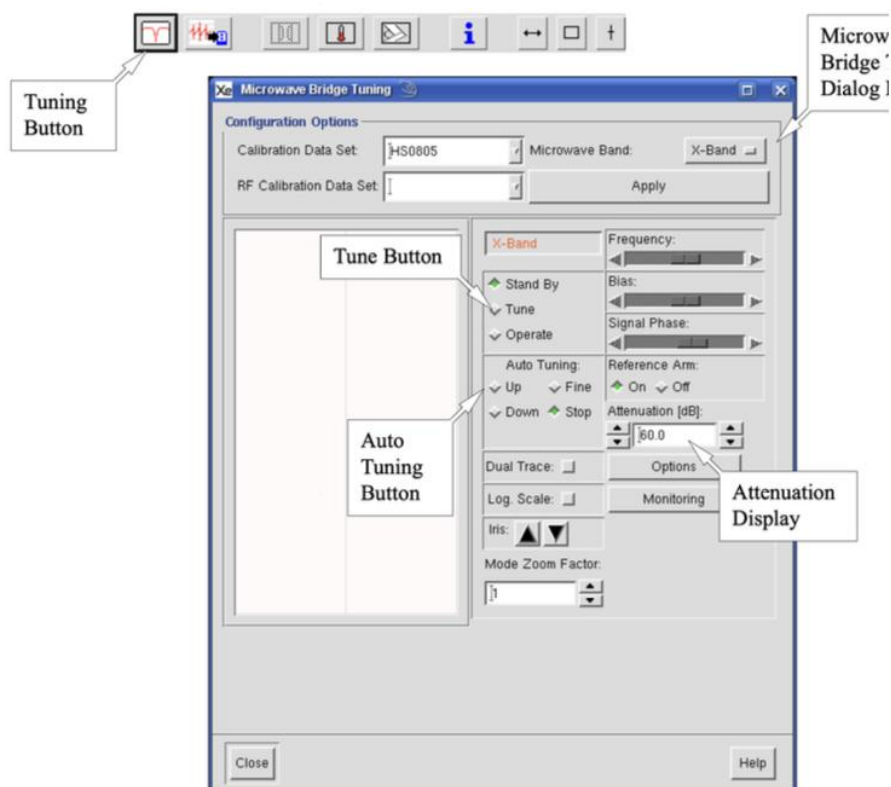


The system should connect within about 30 seconds.

STEP 3. INSERT SAMPLE

Before remove or exchange the samples, you must first open the “Tune window” and exit the operate mode, make sure it goes to the “Stand by” before remove or insert sample.

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1) Take the 4mm OD tube with the black nut out from the top of resonator; Be careful NOT TO bend or drop the tube on the ground.

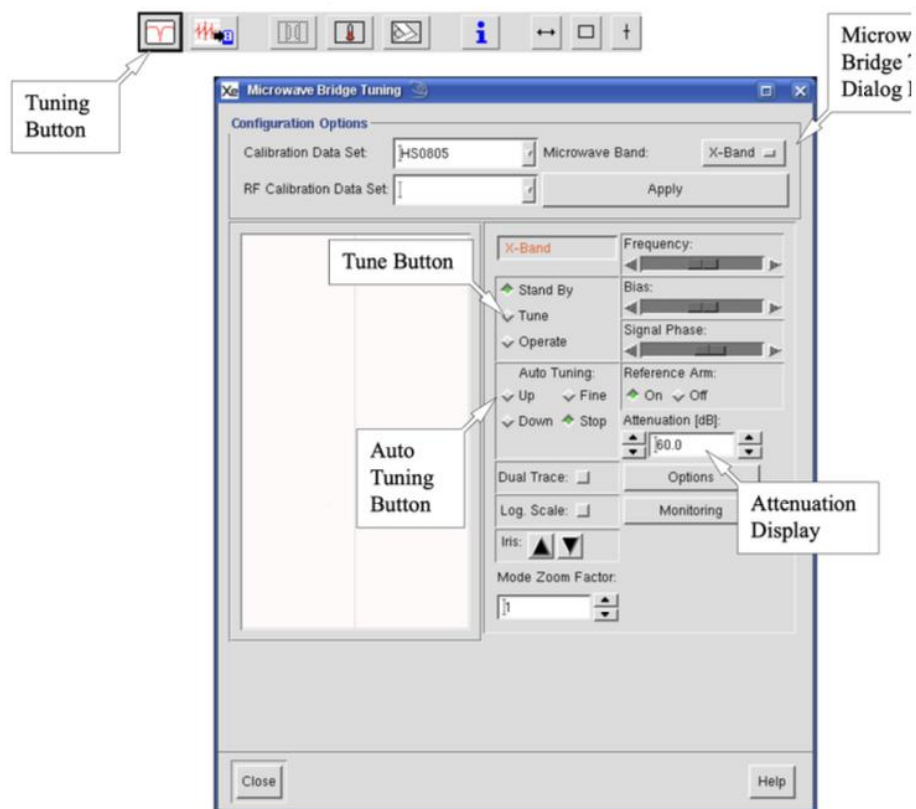
2) Wipe the outside of the tube with acetone or isopropanol. Insert the capillary with the sample into the 4 mm tube. Then insert the whole stuff straight down so do not break the quartz dewar or your sample in the cavity.

STEP 4. TUNING THE SPECTROMETER

1) Click the Tuning button to open the Microwave Bridge Tuning dialog box. The system should

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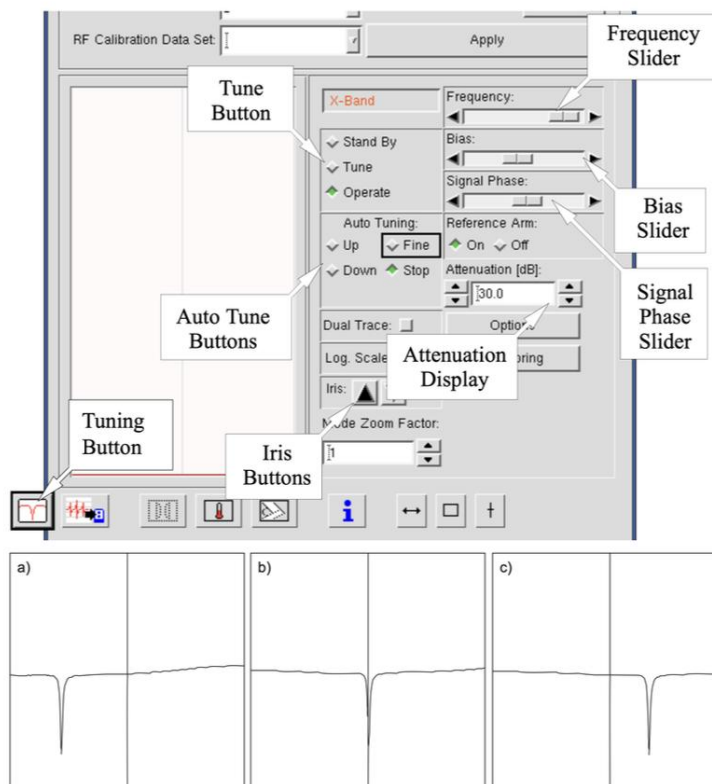
be in Standby mode when opened. The attenuation should be 60 dB.



2) Click the Tune mode...

Set the attenuation to 20 dB. Use the frequency bar to find the dip. If the phase is not good, use the signal phase bar to adjust the symmetry of the dip.

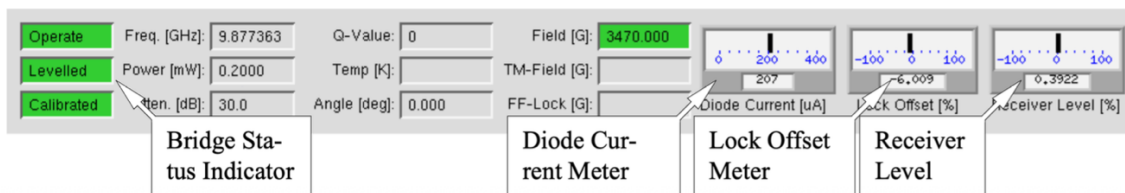
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The click the up button in the auto tuning, the software will start the auto tuning operation. When it is completed successfully, the software will auto switch to the “Operate Mode.”

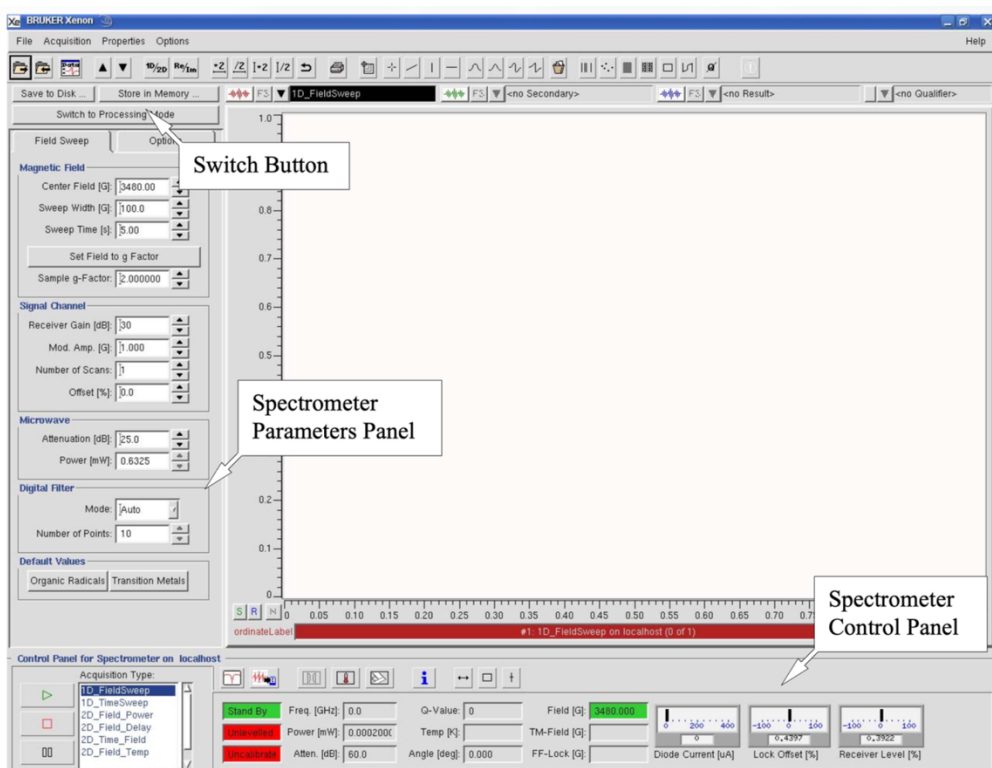
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4) Adjust the Iris up/down arrows  if the diode current meter is not in the center.



STEP 5. COLLECTING A SPECTRA

1. Switch to the acquisition mode by click the button “switch to acquisition mode”



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- change the parameters in the parameters panel.

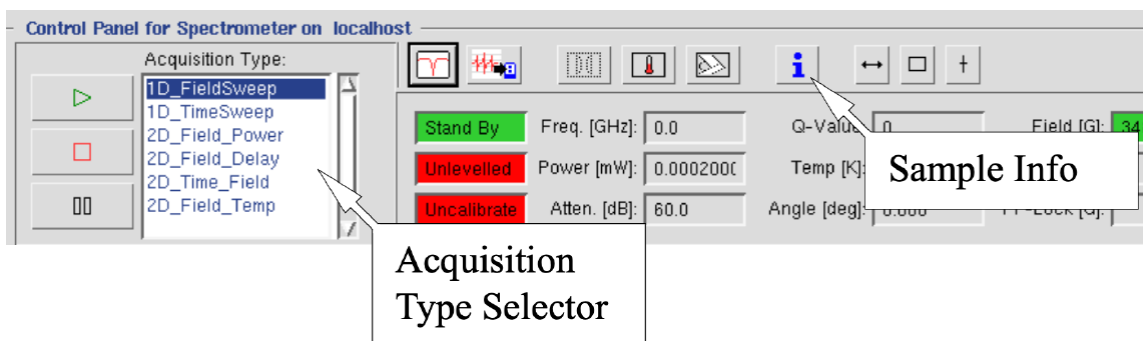
	The Field Sweep Tab The Magnetic field defaults are a good place to start. You can narrow the Sweep Width to save time and you can increase the Sweep Time to improve resolution. The Receiver Gain setting is not changed when you load either default starting parameters. 30 dB is a good starting point. If you see peak tops clipped; lower the gain. Increase if signal is weak. The Modulation Amplitude is one of the more important parameters. Too large and you get line broadening and distortion. Too small and the sensitivity can be poor. If you expect sharp lines leave this set to 1 G. Increase this up to the line width (2-5 G) to improve sensitivity on broader peaks. Signal to noise can also be increased by running multiple scans. 2mW of Microwave Power should be adequate for most samples. Leave attenuation set to 20 dB. Increase attenuation (<i>i.e.</i>, lower power) if peaks are saturated. If possible, we recommend turning off the Digital Filter and smoothing the spectrum in processing mode. Digital filtering here is irreversible, while smoothing after collection can be optimized in a reversible fashion. To turn off Digital filtering set the Mode to "Manual" and the Number of Points to "1". The buttons to load the Default parameter Values
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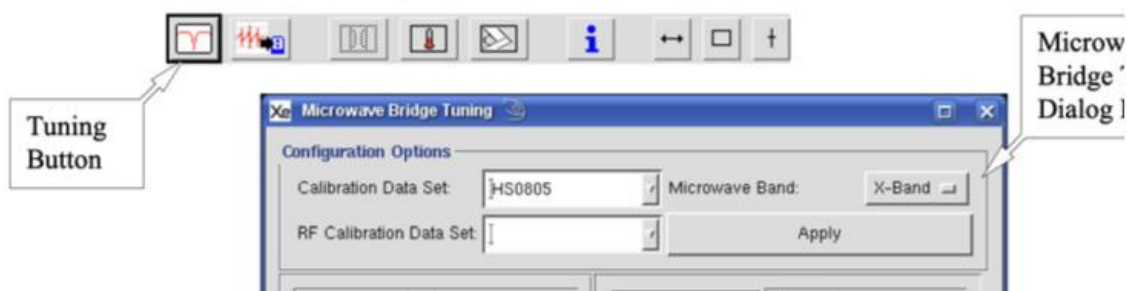
Field Sweep	Options
Magnetic Field Number Of Points: 2000 Field Settling: Wait LED off Settling Delay [s]: 0.0 Sweep Direction: Up Field Flyback: On Signal Channel Calibrated: ☒ Modul. Freq. [kHz]: 100.00 Modulation Phase: 0.0 Conv. Time [ms]: 15.00 Time Constant [ms]: 0.01 Dual Detection: OFF Pts / Mod. Amp.: 10.00 Scan Fine Tune Each Scan: On Off Auto Scaling: On Off Replace Mode: On Off Auto Offset: On Off External Trigger: ☐	The Options Tab The Number of Points, the Conversion time and the Modulation Frequency have a connected relationship. You cannot set the Number of Points directly and it could be a very low number by default. A desired value is close to 1000 and is adjusted by changing the Point per Modulation Amplitude below. The Field Settling is important for experiments collecting multiple scans. The Settling Delay in combination with Field Settling set to "Wait Given Delay" can assure each scan starts at the correct field. Setting Field Settling to "Wait Stable" usually will give good results with multiple scans with minimal wait between scans. A Modulation Frequency of 100 kHz will be adequate for most experiments. The Conversion Time is the time spent at each point and should be left unchanged. The Time Constant is an analog filter setting to help signal to noise. Increasing this value will reduce noise however you can lose fine structure and peak intensity. A safe rule of thumb is to keep the Time Constant much less than the Conversion Time ($\leq 10\%$). The Points per Modulation Amplitude will control the number of points collected. Set this so the Number of Points is between 1000 -1200. The Fine Tune Each Scan is very helpful for large number of scans (long acquisition). It will allow the instrument to touch up the cavity tuning to compensate for any instrument drift that occurs during the experiment.

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3. The system has several acquisition type, make sure that you select the right one. For instance, normal 1D will chose the 1D_fieldsweep.



4. Open the tuning bridge window, make sure the right resonator is chosen. This step is usually configured by the staff, but better check it.



If forget this step, then when run the first experiment it will say “uncalibrated” in red letters at the top of your spectrum.

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5. Click Run

STEP 6. SAVING THE DATA

File => Save as... => My Computer => EPR Data => User's name folder

** The software saves two files: one for the spectra and one for the parameters. Be sure that if you move one, you move the other by clicking on All Files from the drop down menu.

As an ASCII file

File => Export... => Destination (notice the file type is ASCII format)

Open this file in Excel and it is delimited with tabs; format as necessary.

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ENDING WORK

- (1) Open the MW bridge window, change it to the “standby mode”
- (2) Move your sample and put the empty 4 mm tube and black holder back to the resonator
- (3) In the acquisition Menu, chose disconnect from Spectrometer, then click “ok”
- (3) Close the software
- (4) Turn off the magnet
- (5) Turn off the console by press the front button.
- (5) Keep desk top clean
- (6) Logout from NUcore

REVISIONS

Use was made of Bruker EMXplus CW X-band at Northwestern University, which has received support from National Science Foundation (CHE-9871268) and International Institute for Nanotechnology (IIN).

V 1.0 2026/02/22	Release of original version of IMSERC CW X-band V1.0
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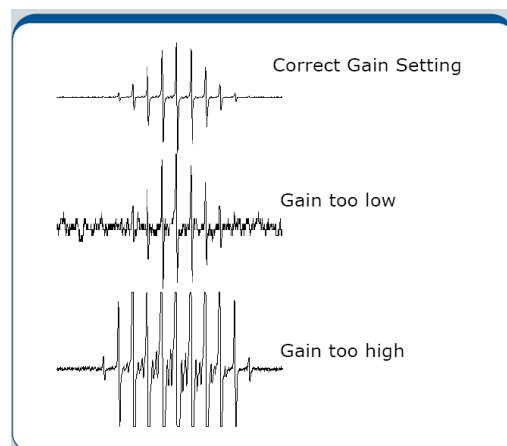
APPENDIX A: HOW TO OPTIMIZE THE PARAMETERS

1) **Center Field and Sweep Width** – Center field should be set at the center of your EPR signal and the sweep width should include the signal and enough baseline. Once you can see your signal you can click on the “Change center field and sweep width” icon in order to set the appropriate values. Size the box to the desired size by clicking with the left mouse button and dragging at each corner. Once you have your desired size set it by clicking the right mouse button then RERUN your spectrum. The new values WILL NOT be saved until you rerun the experiment.

2) **Receiver Gain** – Depends on the strength of the EPR signal from your sample. If your signal is saturated lower the Receiver Gain.

Strong Signal = Low R.G.

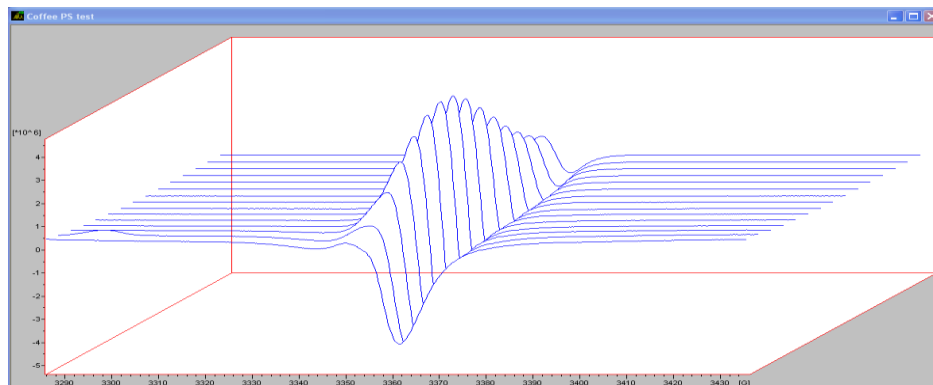
Weak Signal = High R.G.



3) **Power Level**

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Once acquisition is finished, right-click on the spectrum and go to *Display Type* => *2D Stack Plot*. Right click again and check *Whitewash* (this shows only positive values). The spectrum with the highest intensity is your optimal power.



4) **Modulation Amplitude** – Helps decrease low frequency noise (i.e. thermal fluctuations, electronic vibrations, drafts in the room, etc.)

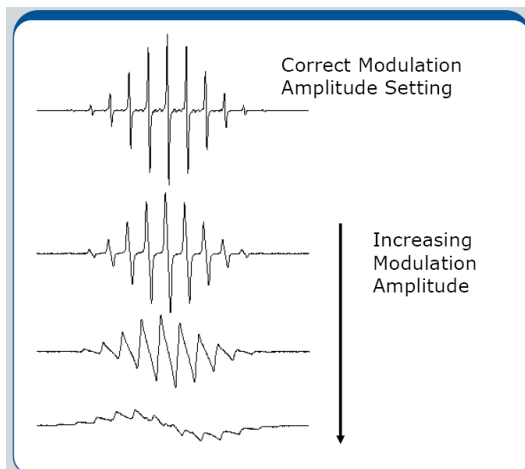
→ Compromise between sensitivity and resolution

→ If too large => Will distort/broaden signal

If too small => Disadvantage because S/N will not be as good

→ This value is sample dependent

* The more hyperfine splitting, the smaller the value should be



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5) **Time Constant** – filters out high frequency noise (slows down the response time of the spectrometer)

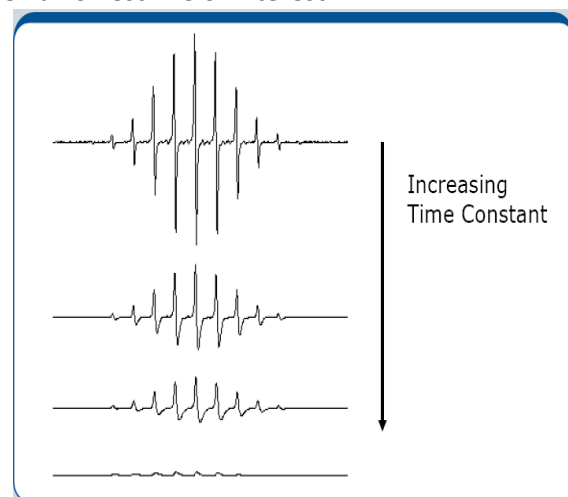
→ Set to 1/10 the time it takes to sweep through the narrowest line of interest

→ Too Low => Get a lot of noise

→ Too High => Filters out your EPR signal

**Want to increase for weak signals. If a long Time Constant is used use a slower scan rate.

**For very narrow lines increase the Time Constant by 2 and increase the number of data points (Resolution in X).

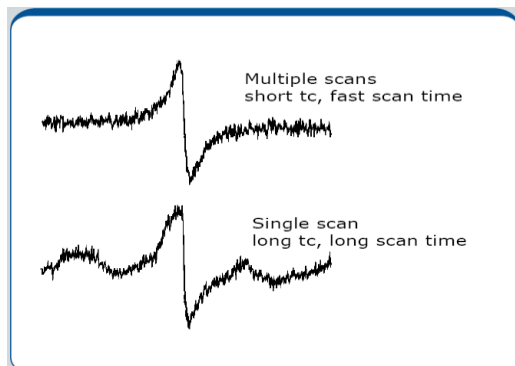


6.) **Conversion Time** – The amount of time the AC to DC converter spends at each point.

→ Rule of Thumb: Set = to the Time Constant

7.) **Number of X-Scans (i.e. Signal Averaging):** The baseline quality of your spectrum is improved by accumulating several short scans rather than taking one long scan.

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APPENDIX B: VARIABLE TEMPERATURE EXPERIMENT

If you need use VTU 3 for variable temperature, please contact Dr. Jinlei Cui for additional training.

Low temperature is necessary for EPR to enlengthen the spin-lattice relaxation time of electron, enhance the signal intensity and freeze the dynamics of electron. Currently, the VTU 3 is connected with the loop resonator, which includes controller, finger-dewar and LN₂ tank. It enables the experiment temperature from 100k to 300k. To do the VTU experiment, please do the following steps.



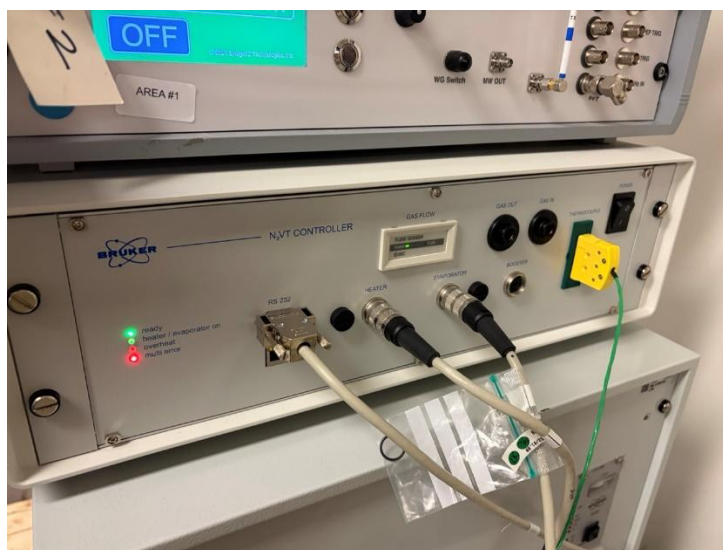
Portable VTU Station

1. VTU system for EMXPlus

(1) fill the LN2 tank in the cryogen dock station

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(2) turn on the VTU controller and turn on the computer. if the LN2 tank is empty, it will show multi errors. The errors will disappear when LN2 is refilled.



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(3) slowly re-cap the LN2 tank with the transfer-line. The LN2 will boil off quickly in the beginning.
(do not forget the o-ring)



(4) connect the figure dewar with LN2 tank transfer line.



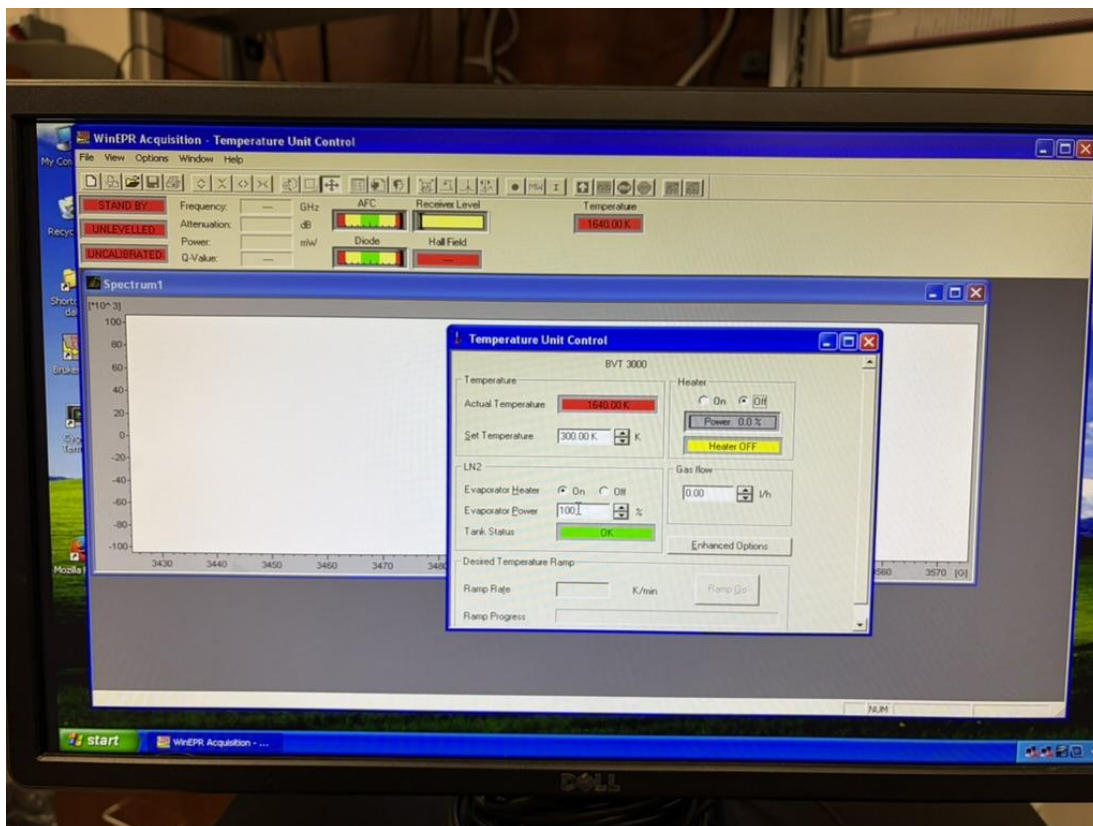
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(5) Connect the heater line and thermal couple line in the VTU controller.



(6) Open the software “Bruker EMX”, in the “file” tab, click the “new experiment”. Then open “Temperature control unit” in the “view” tab. (ignore the errors).

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(7) Click “on” in the evaporator “heater”, input “100” in the evaporator power.

(8) turn on of the “heater”

(9) set the temperature in the interface (such as 100k). For different temperature, you can adjust the evaporator power. For example, for higher sample temperature, lower evaporator power should be used. But the lowest evaporator power is 25% for stable temperature controlling.

(10) When the temperature is stable, tuning the resonator for your exp.

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(11) when VT experiment is done, set the temperature to 295k, then set the LN2 evaporator heater power to 50, wait until the resonator is totally warm (usually 15 mins).

(12) Change the LN2 evaporator heater power to 0, change the LN2 evaporator heater to off, change heater to off.

(13) close the variable temperature unit.

(14) disconnect the “heater” and “thermal couple” cable from the VTU controller.

(15) remove the transfer line from LN2 tank

(16) turn off all the instruments and relocated them to original location.

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APPENDIX C: ULTRA LOW TEMPERATURE – ESR900

The ESR900 could cool down the sample to 4k. If you need to use this, please email Dr. Jinlei Cui for at least three days ago. Dr. Cui will set up the ESR900 and teach you how to change sample. That requires a additional training.

Change samples for the ESR900 using the following steps:

1. Turn off the pump.



2. The He gas flow in the exhaust flow meter will go to zero. However, there may still be some residual gas flow or pump flow.

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3. Remove the cap from the top of the ESR900.
4. Quickly transfer the pre-cooled sample tube with the sample holder into the ESR900.
Note: Leave enough space for the sample tube. First, insert the sample and seal the sample holder. Then insert the EPR tube into the Teflon positioner and seal it. This way will ensure the EPR tube does not damage the supporting tube inside the ESR900.



5. Turn on the pump.

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