Bruker EMX EPR User's Booklet

If you are having problems with the instrument, or have any questions, please contact Jim Windak, room 3411b Chemistry, phone # 647-2847, e-mail: jwindak@umich.edu.

I. Turning on the Instrument

1) Turn on the water chiller unit located in back of the magnet.

On / Off Switch for the water chiller.

2) If you will be doing low temperature helium work, turn on the nitrogen gas valve for the cavity purge gas line. This is the valve located on the wall on the right side. This directs a small flow of dry nitrogen gas inside of the EPR cavity in order to prevent moisture condensation. If the red handle is parallel to the pipe, the flow is on. If it is perpendicular to the pipe, the flow is off.

If you are using the nitrogen cryostat, you do not need to do this. The nitrogen purge comes from a different source when using the nitrogen cryostat.

Turn this handle up only when doing helium EPR.

3) Turn on the “Electric On” button located on the front of the magnet power supply. The five LED’s located on the front will all turn on, and then they should all turn off after approx. 20 seconds. If they do not all turn off, then there is a problem, and you should please contact me. After all of the LED’s turn off, you should press the green button to turn on the magnet current.

Press the Electric On button on first.

After the 5 LED’s turn off, push the green ON button.

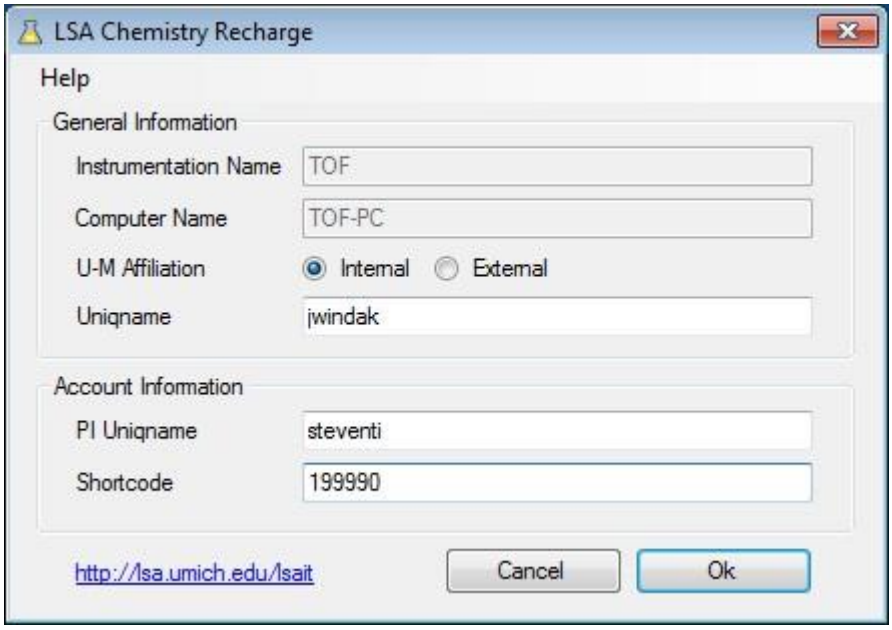
These 5 LED’s will turn on for 20 seconds, and then turn off.

4) Turn on the power switch for the instrument electronics, located on top of the electronics module.

On / Off Switch for Electronics

1) Double-click on the LSA Chemistry Recharge icon:

Type in your uniquename, your PI's uniquename, and your short-code account number and click Ok:

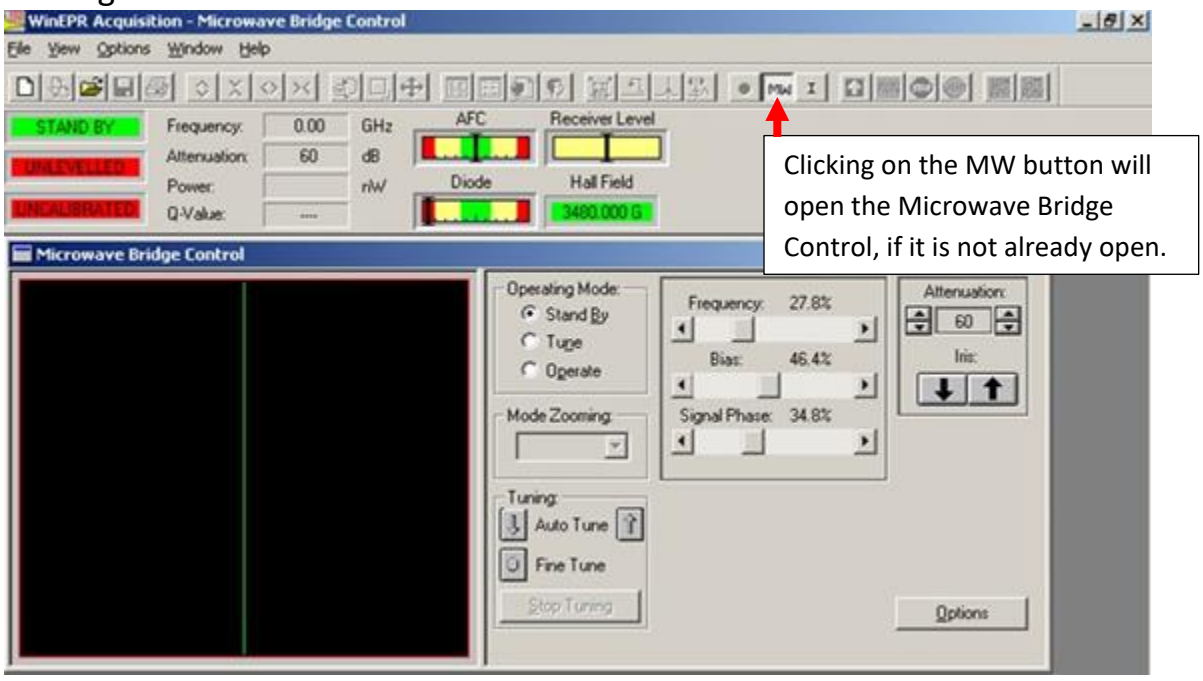


The WinEPR data acquisition software will then automatically come up.

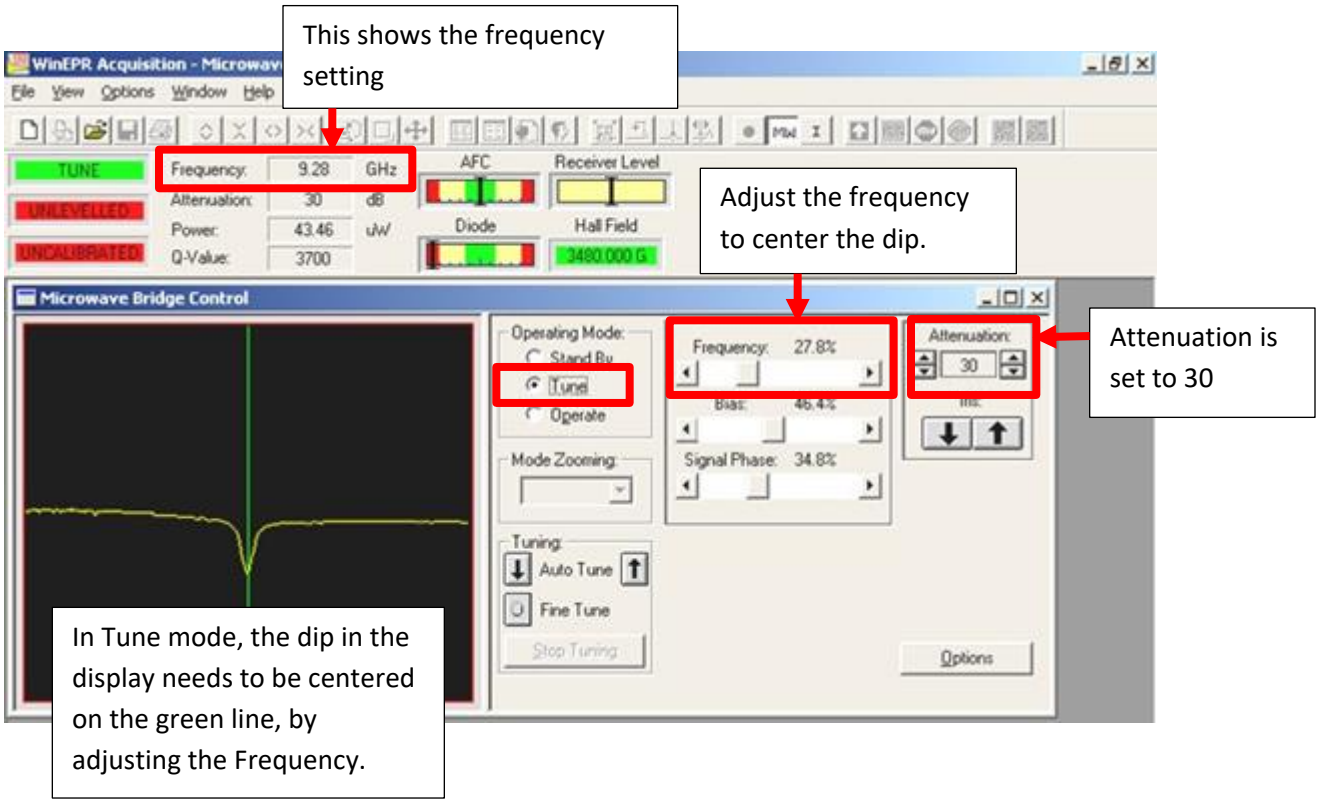
II. Tuning the Cavity

Before you run each sample, you must tune the cavity.

1) If the Microwave Bridge Control dialog box is not already open, you can open it by clicking on the "MW" button on the toolbar.



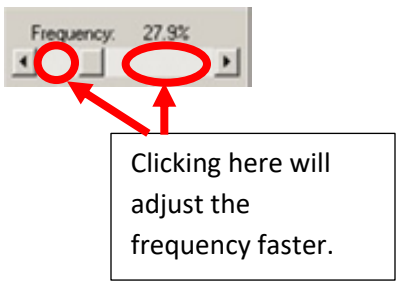
2) Click on the Tune button, and lower the attenuation setting to 30. You should see a dip in the display. If you do not, then you need to adjust the microwave frequency so that you see the dip in the middle of the display. As a guide, an empty cavity will tune around 9.78 Ghz. A cavity with the Helium cryostat installed will tune around 9.45 Ghz, and a cavity with the nitrogen gas cryostat installed will tune around 9.28 Ghz.
Adjust the frequency so that the dip is exactly centered on the green line.



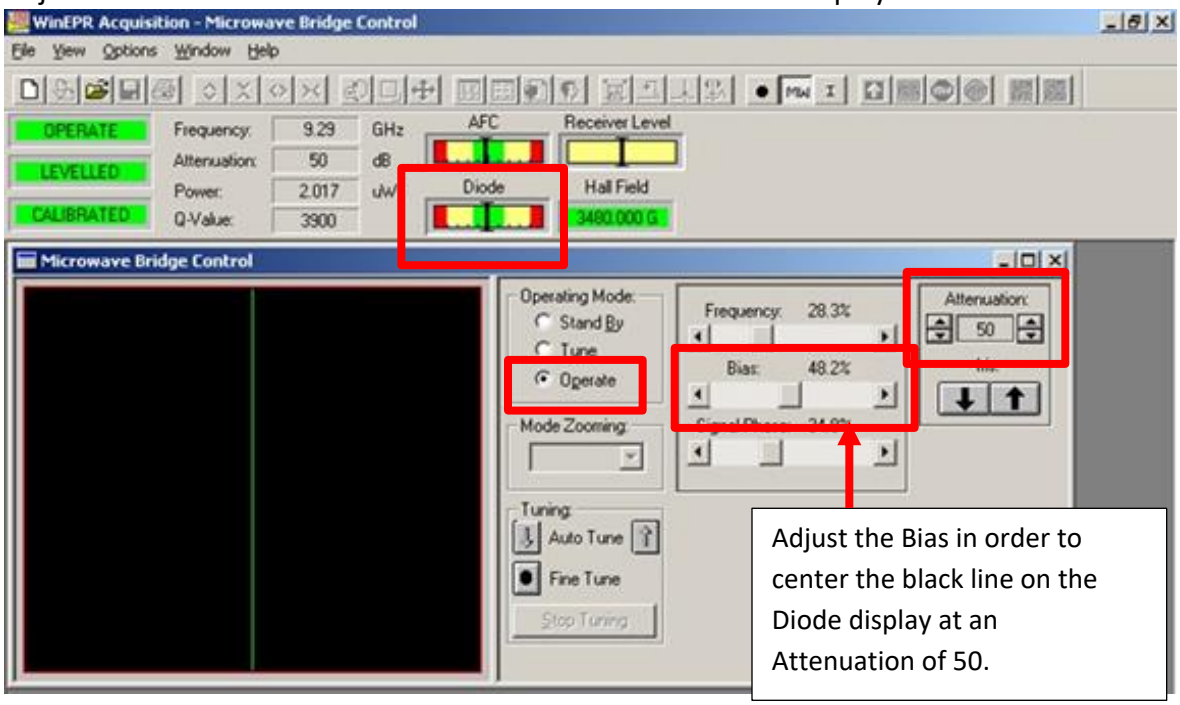
Please note:

When adjusting the Frequency, clicking on the ◂ and ▸ buttons will adjust the frequency slowly.

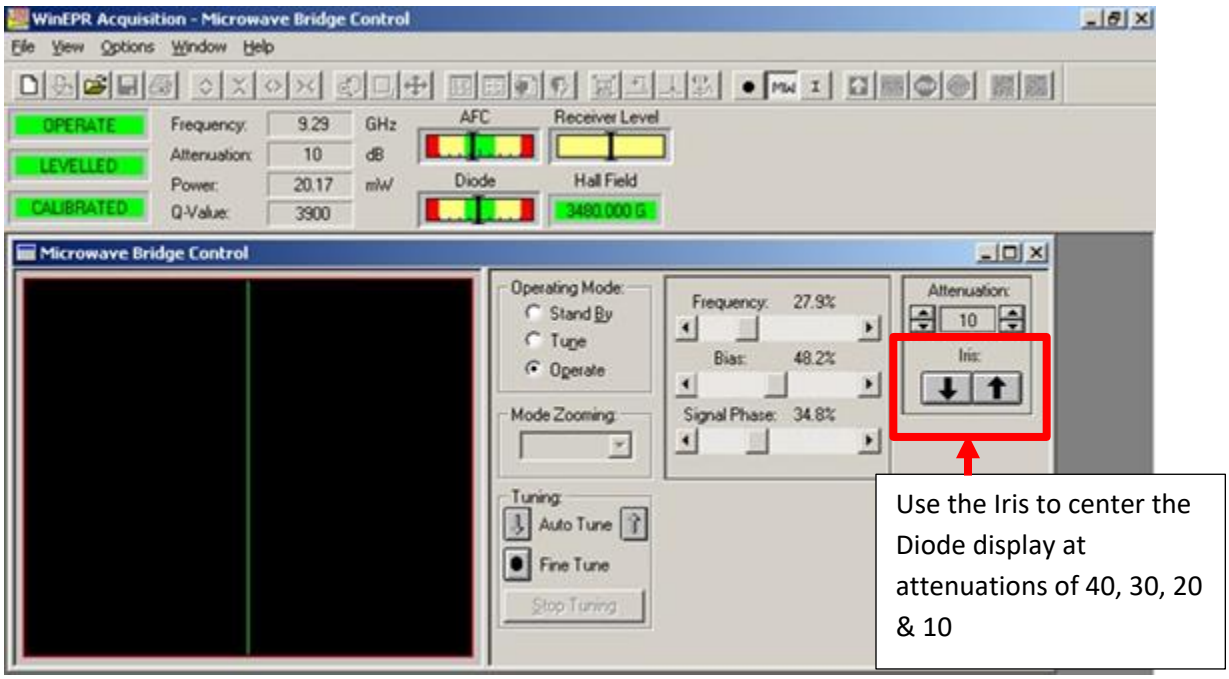
Clicking on the space in the slider will adjust the frequency faster.



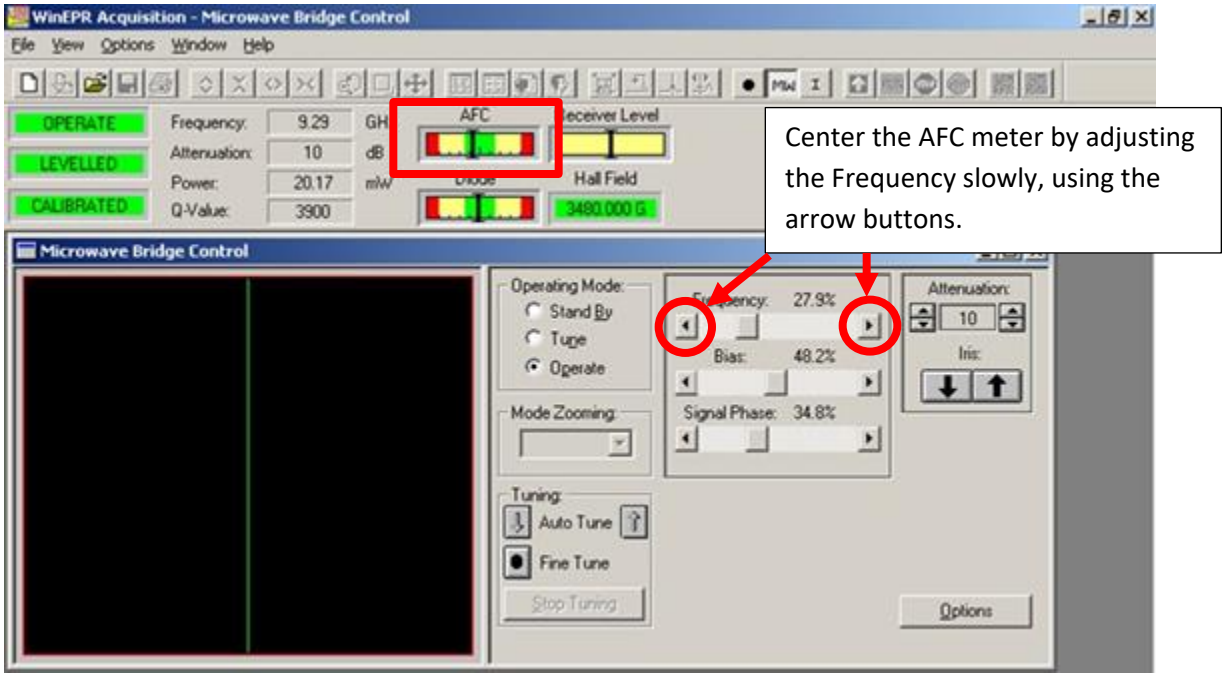
3) Next, click on Operate.
Set the Attenuation to 50.
Adjust the Bias in order to center the black line on the Diode display.



4) Decrease the Attenuation to 40.
If the Diode meter is not centered, center it by adjusting the **Iris**. Do not center it by adjusting the bias. The bias is used to center the Diode meter *only* at an attenuation of 50, and at no other time. At all other attenuations, the diode meter is centered by adjusting the Iris.
Repeat this procedure at an Attenuation of 30, then 20, and then 10.



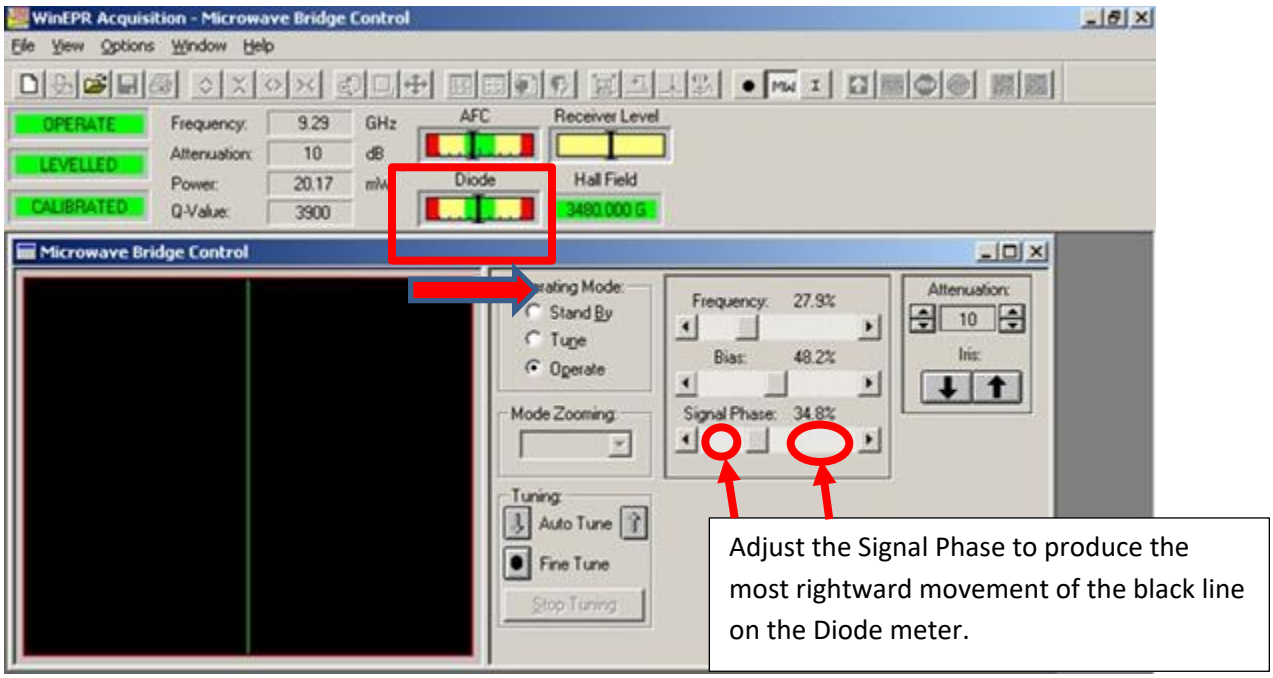
5) Center the black line in the AFC meter by adjusting the Frequency very slowly, using the ◄ and ► buttons.



6) Fine-tune the signal phase, while at an attenuation of 10. This step only needs to be done once at the beginning of your session. After that, it should remain stable.

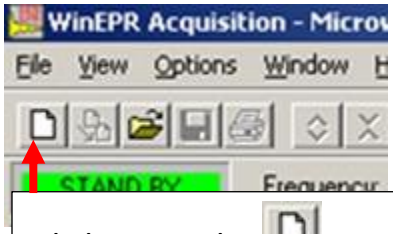
To do this, adjust the Signal Phase control by clicking in the space inside of the Signal Phase slider bar. Set it to where it will produce the largest (most rightward) movement of the black line on the Diode meter.

Then, after making this adjustment, if the Diode meter is not centered, re-center it by adjusting the Iris.

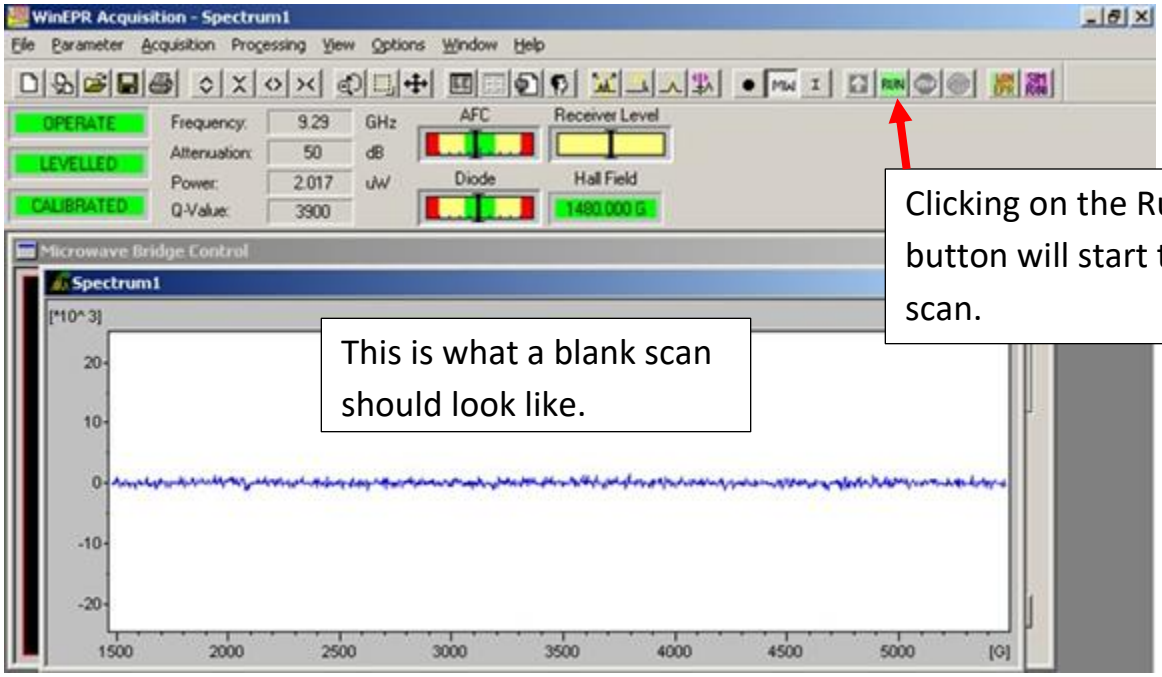


III. Running a Blank Spectrum

Run a blank to show that the cavity is clean was not contaminated by the previous user. For the blank run, the attenuation should be set to 10. The default acquisition parameters should be used. Click on the spectrum window to make the window active, and then click the Run button. If no spectrum window is present, click on the  icon on the toolbar.



Clicking on the  icon will bring up a blank spectrum window, with default acquisition parameters.



Clicking on the Run button will start the scan.

This is what a blank scan should look like.

After the scan is completed, go back to the Microwave Bridge Control box, and set the attenuation to 60. Then click on Standby.

Please note: You must always set the attenuation to 60, before going to Standby.

IV. Running Samples

Please note: You must be extremely careful to avoid contaminating the cavity. Clean the outside of your sample tube with alcohol and Kim Wipes to make certain it is clean before inserting it into the cavity.

Please note: Be certain that the microwave bridge is in the Standby mode whenever you are inserting or removing samples.

Please note that if you are running aqueous samples, the sample must either be frozen, or else if not frozen, then it must be run in a special flat cell in a TM-mode cavity. You cannot tune a cavity containing a standard 4 mm O.D. tube filled with liquid water. This also applies to other polar solvents.

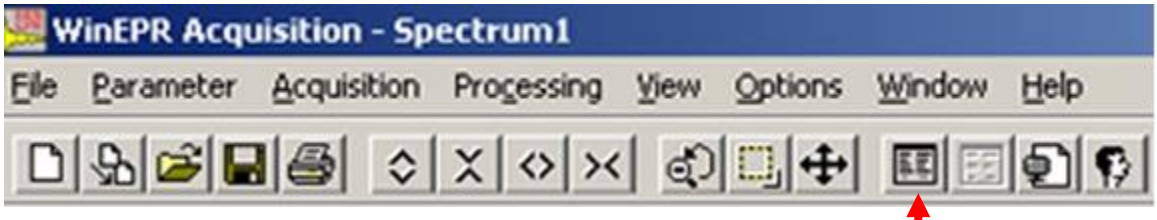
If you are using the nitrogen gas cryostat, insert the sample so that it is centered in the middle of the resonator cavity, and then snug down the collet fitting at the top.

Use high quality 4 mm O.D. quartz tubes. Lower quality quartz may exhibit a small EPR background signal.

V. Setting Acquisition Parameters

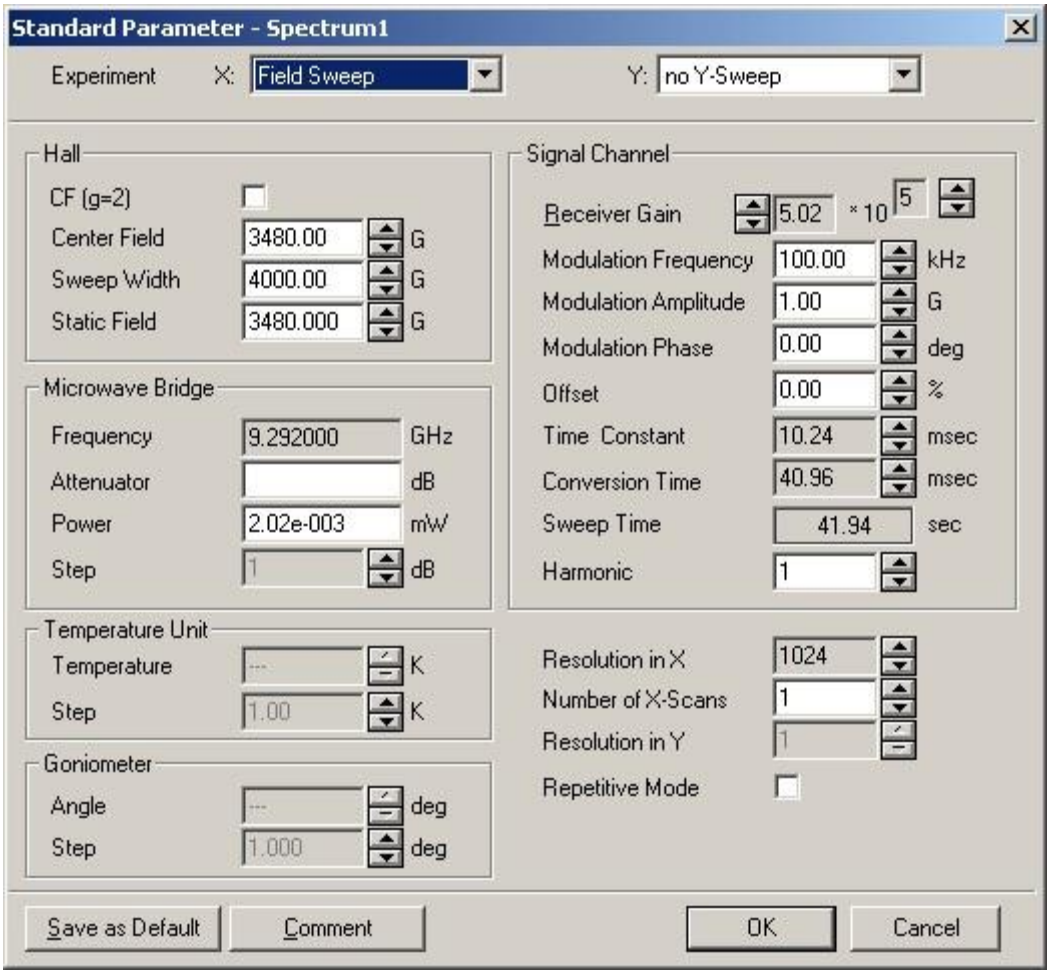
When you click on the  icon on the far left of the toolbar, you will get a new spectrum window with the instrument acquisition parameters set to their default values. These are the parameters that we use for collecting blank scans both before and after using the EPR to check for cavity contamination.

You will need to change some of these parameters to suit your particular experiment. Do this by clicking on the Experiment Parameters button on the toolbar.



Clicking on this button will let you change data acquisition parameters.

This is the parameters window:

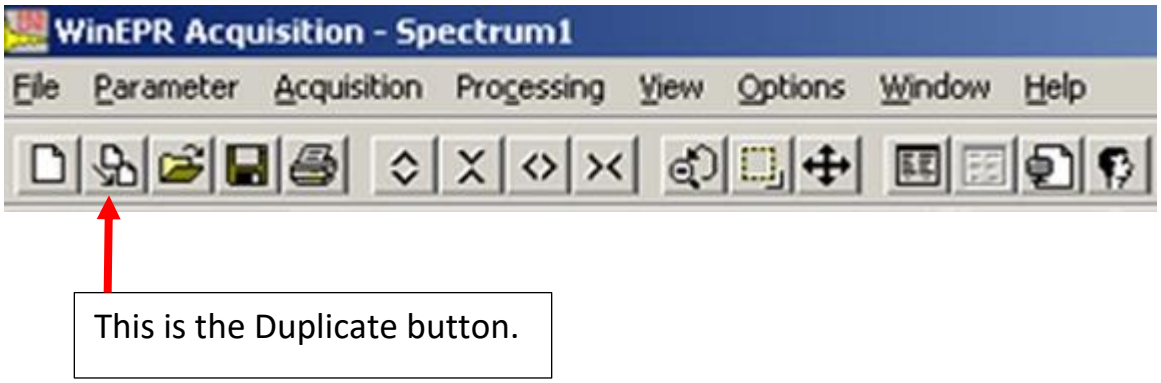


Some parameters you may need to change:

Center Field:	Sets the midpoint of the scan in gauss.
Sweep Width:	Sets the total length of the scan in gauss.
Receiver Gain:	Sets the amplification of the EPR signal. Increasing the Gain will increase the size of your signal, but if set too high the signal will be clipped off.
Modulation Amplitude:	Increasing this parameter will increase the size of weak signals, but it also increases the distortion of the signal. **Please do not exceed 32 gauss of modulation amplitude for a 4102-ST cavity, and do not exceed 16 gauss for a 4103-TM cavity. Damage to the cavity will result!**
Time Constant:	This controls electronic filtering of noise. Making this value large will filter more noise, but it will also cause signal distortion if set too high. This should not be set higher than the conversion time.

Conversion Time:	This is the time in milliseconds that is spent on each data point. Higher conversion times give better results on noisy and weak signals, but will increase the scan time.
Resolution in X:	This is the number of data points across the total scan. The Conversion Time and the Resolution in X both affect the total scan time.
Number of Scans:	This will determine how many scans are added together.

Helpful Tip: Once you have set your acquisition parameters for one sample, you do not need to reset them for each sample if you use the duplicate button  on the toolbar. If you select an acquisition window and click the duplicate button, you will get a new acquisition window with the same instrument parameters that were in effect for the old window.



Please note that when you acquire an EPR spectrum, it is not automatically saved.
If you want to permanently save the data, you need to click on the spectrum window to highlight it. Then do a File, Save As… This will save the spectrum is a format that is readable by the Bruker WinEPR software.

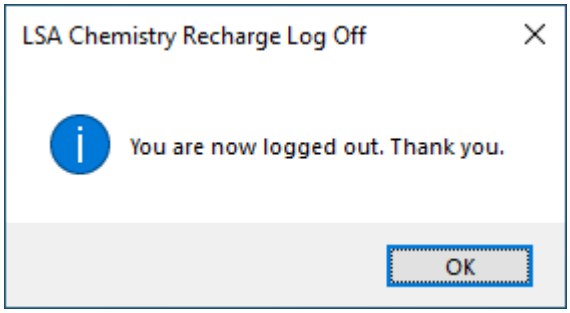
You can also export the spectrum as a text file listing the raw data points.
Do this by clicking on File and Export…

VI. Shutting Down the Instrument

1) Run a blank scan to make certain that you have not contaminated the cavity, using the standard default conditions. Please put your name on it and print it out, and put the printout into the tray by the printer.

2) Raise the attenuation to 60; put the microwave bridge into the standby mode.

3) **Be sure to close the WinEPR data acquisition software. This will close out your account so that you will not be billed for any more time. After the software closes, you should see this message:**



4) Turn off the switch on top of the electronics console.

5) Turn off the magnet current using the red button on the magnet current power supply. Then turn off the "Electric On" button.

6) Turn off the water chiller unit.

7) Turn off the nitrogen gas, if you were using nitrogen gas for the cavity purge when doing liquid helium experiments.

   If you were doing liquid nitrogen experiments, turn off the liquid nitrogen controller.