

ESR User's Booklet for the Bruker EMX

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 water chiller

- 2) If you will be doing low temperature 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.

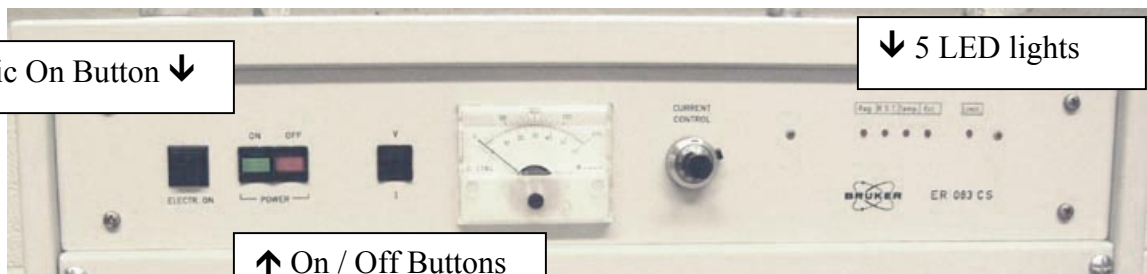
Valve for nitrogen cryostat →



← Valve for nitrogen cavity purge

- 3) If you will be using the nitrogen gas cryostat, turn on the nitrogen gas supply for the cryostat. This is the valve located on the wall on the left side.
- 4) 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.

Electric On Button ↓



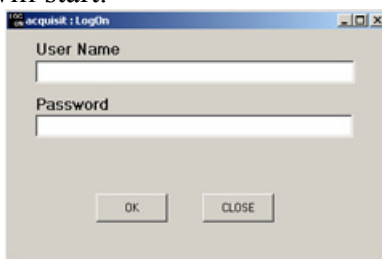
↓ 5 LED lights

↑ On / Off Buttons for magnet current

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

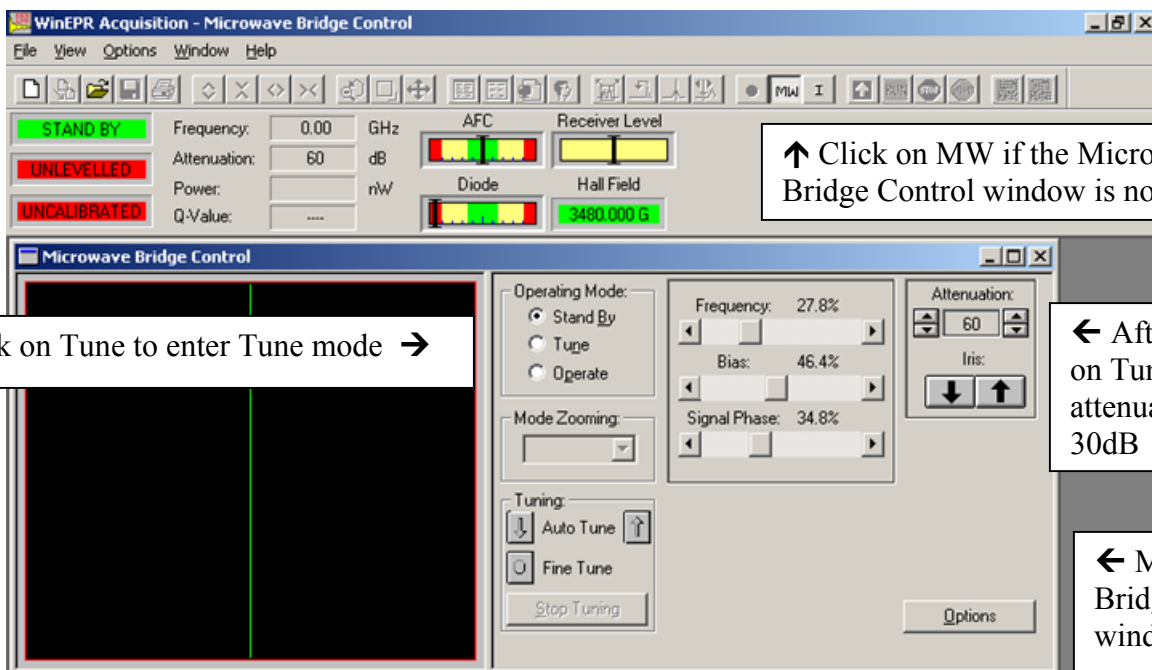


- 6) Wake up the computer by moving the mouse. After the computer wakes up, you can log in. If you do not see the login box, double-click the WinEPR Acquisition icon and it will appear. After you log in, the Acquisition software will start.



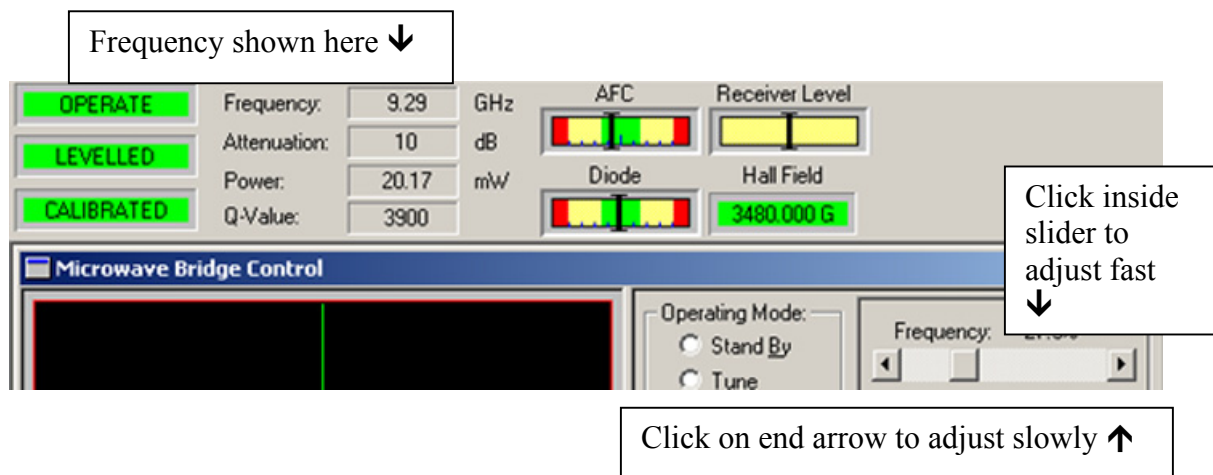
II. Tuning the Cavity

- 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.

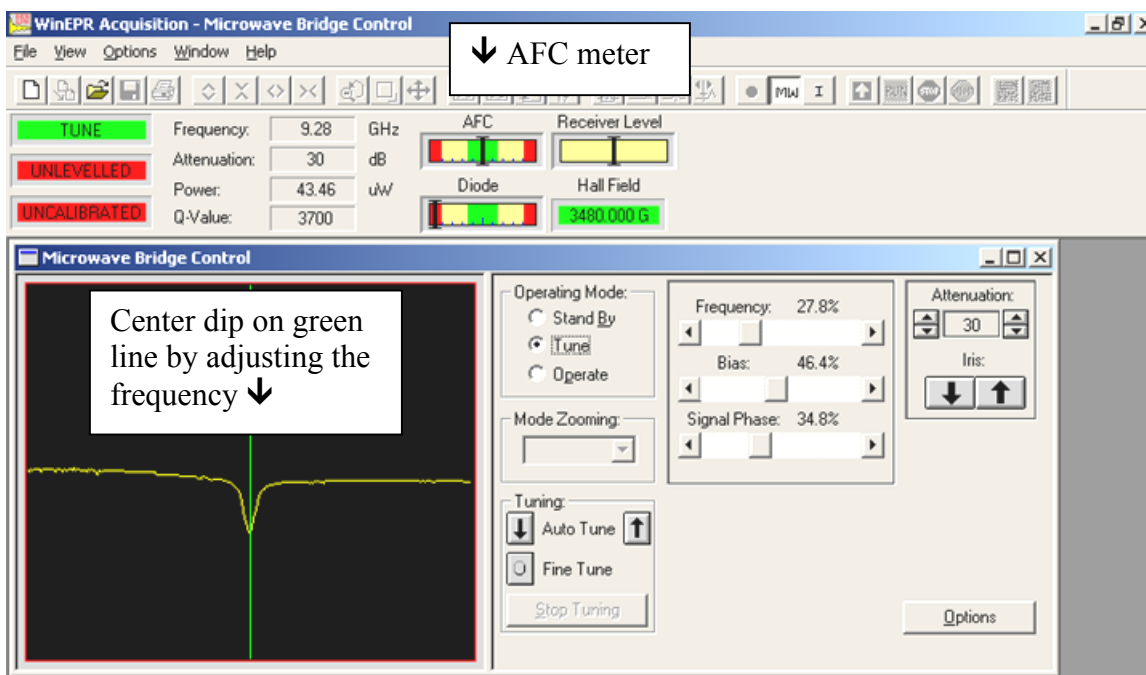


- 2) Click on the Tune button, and lower the attenuation setting to 30. You should see a dip in the display. If you do not, then you need to adjust the microwave frequency so that you see the dip in the middle of the display. As a guide, you should know that an empty cavity will tune around 9.78 Ghz. 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 9.26 Ghz.

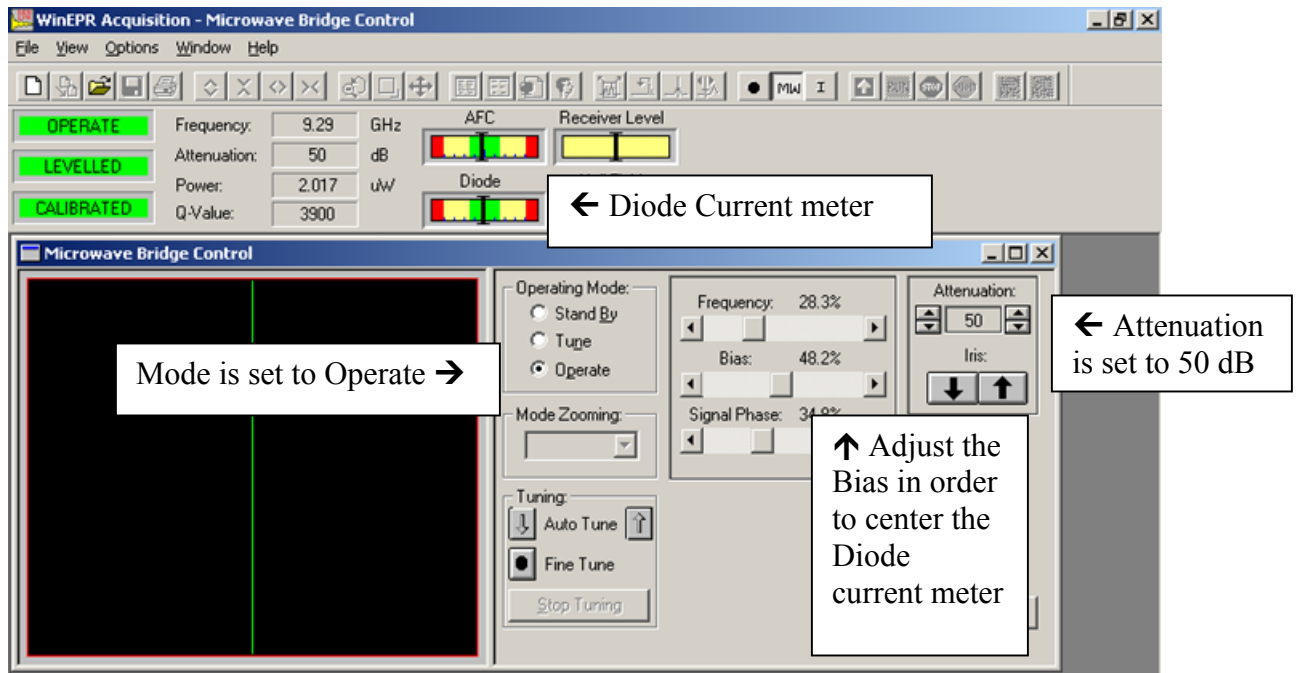
Please note: To adjust the frequency slowly, you click on the arrow boxes on either end of the frequency adjustment slider. To adjust the frequency quickly, click on the space inside of the slider. Unless you are way off from the proper value, you generally want to adjust the frequency slowly, especially when you are in the operate mode. In the operate mode, you adjust the frequency very slightly by making only single clicks on the arrow boxes.



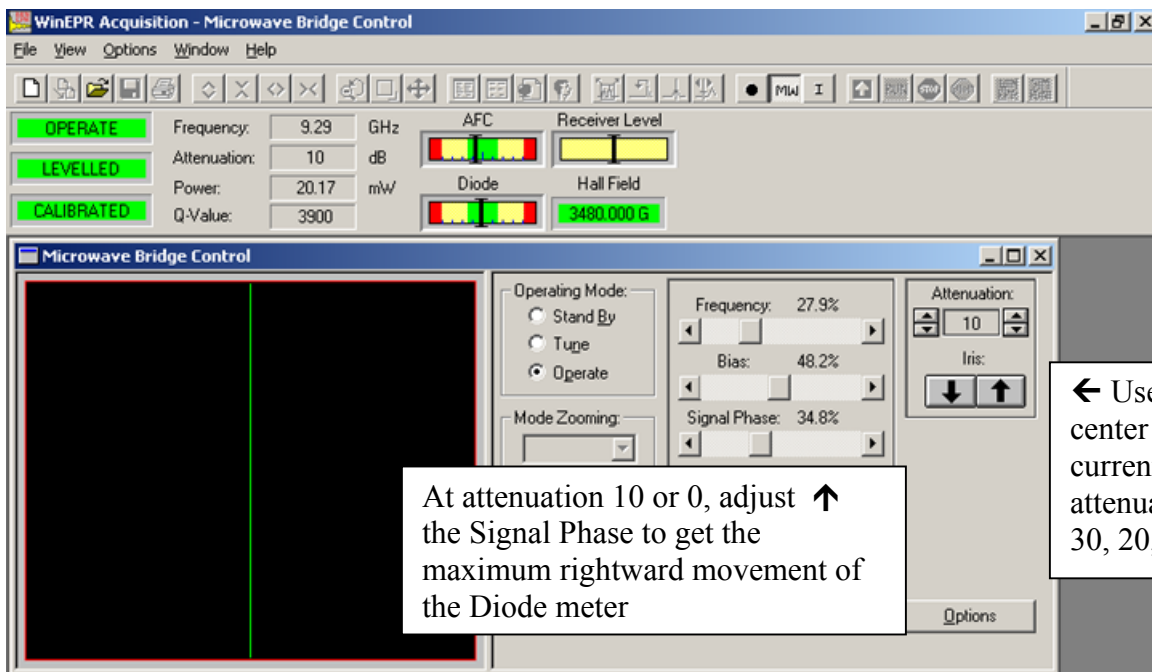
- 3) Center the dip in the display by adjusting the Frequency. Then make the line flat on either side of the dip by adjusting the Signal Phase slider.



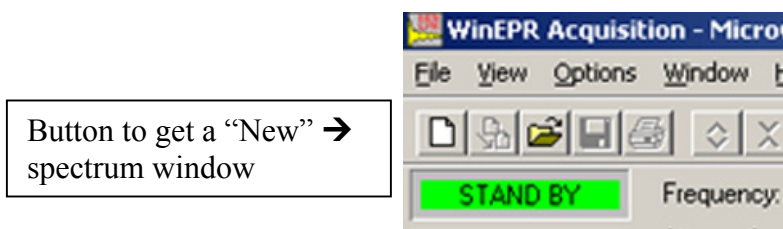
- 4) Now click on Operate. Look at the AFC meter. If it is off scale, then you need to go back to Tune, and try to move the dip closer to the center of the display. Then try to go to Operate again. If the AFC meter is not off-scale (in the red zone), then you are ok. Now adjust the frequency very slowly with single clicks on the arrow boxes in order to position the AFC meter indication exactly in the center of the meter.
- 5) Now increase the attenuation to 50. Re-center the AFC meter if necessary using the frequency slider. Now adjust the Bias, in order to center the diode current meter.



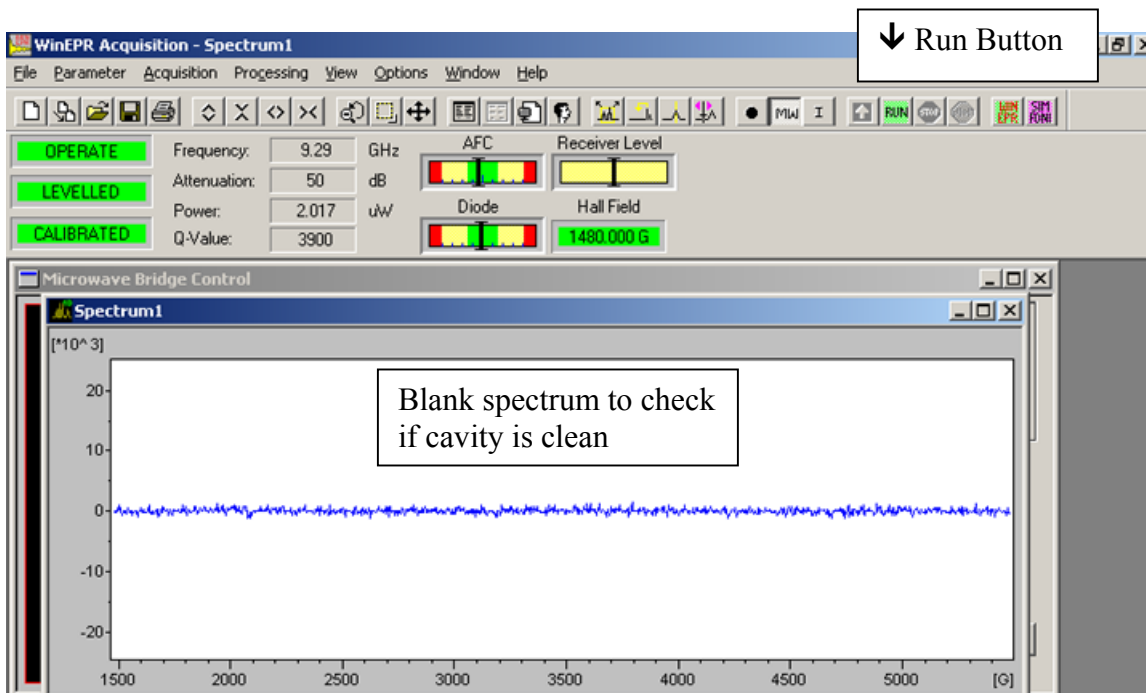
- 6) Decrease the attenuation to 40. Re-center the AFC meter if necessary using slight frequency adjustments. Now if the diode current meter is not centered, you center it by adjusting the Iris. Do not center it by adjusting the bias. The bias is used to center the diode current meter *only* at an attenuation of 50, and at no other time. At all other attenuations, the diode current meter is centered by adjusting the Iris.
- 7) Decrease the attenuation to 30. Repeat the process by adjusting the frequency slightly to center the AFC meter, and adjusting the Iris slightly to center the diode current meter.
- 8) Repeat this same process at attenuations of 20, 10, and 0.



- 9) Now we need to fine-tune the signal phase, while at an attenuation of 10 or 0. Adjust the signal phase control by clicking in the space inside of the slider bar (this is a more coarse adjustment than the frequency). You want to set it to where it gives the largest (most rightward) display on the diode current meter. Then, after making this adjustment, if the diode current meter is not centered, you need to re-center it by adjusting the Iris.
- 10) Now you should have the cavity properly tuned. To check this, adjust the attenuation in steps of 10 from 0 to 50. At each step, the AFC meter and the diode current meter should both be fairly closely centered.
- 11) 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 New icon on the toolbar.



After the blank run has been acquired, click on the person icon, and type in your name under user. Under comment, type "blank run before". Scale the y-axis to +10,000 to -10,000 units, by clicking on View, then Scaling, and then Manual, and then enter these values into the dialog box. Then print out this spectrum and put it into the tray next to the printer.



- 12) 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.

III. 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 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.

If you are not using a cryostat, then your sample tube must be secured using a Teflon collet that matches the diameter of your sample tube. Insert your sample so that it is centered vertically in the middle of the cavity, and then snug down the collet but do not overtighten it.

If you are using the nitrogen gas cryostat, then gently insert the sample until it bottoms out on the indentations in the quartz insert, and snug down the collet fitting at the top.

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

Do not overfill your sample tube, especially if you are using a cryostat. Typically, do not exceed 2 or 3 cm of sample height in the tube. Use high quality 4 mm O.D. quartz tubes. Lower quality quartz may exhibit a small EPR background signal.

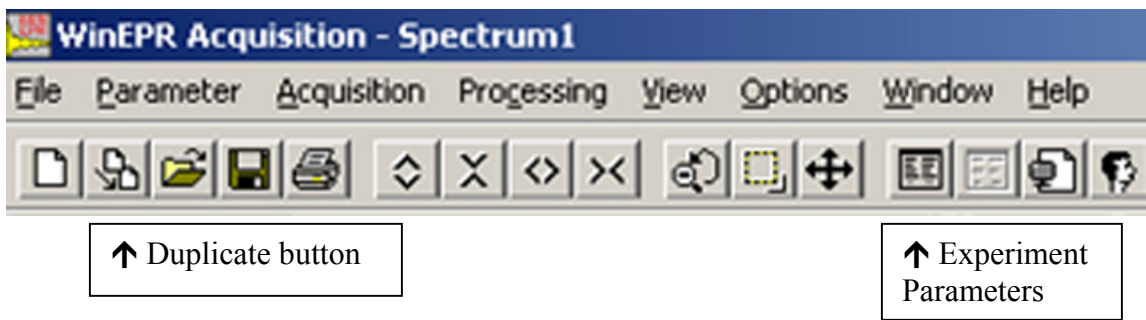
IV. Setting Acquisition Parameters

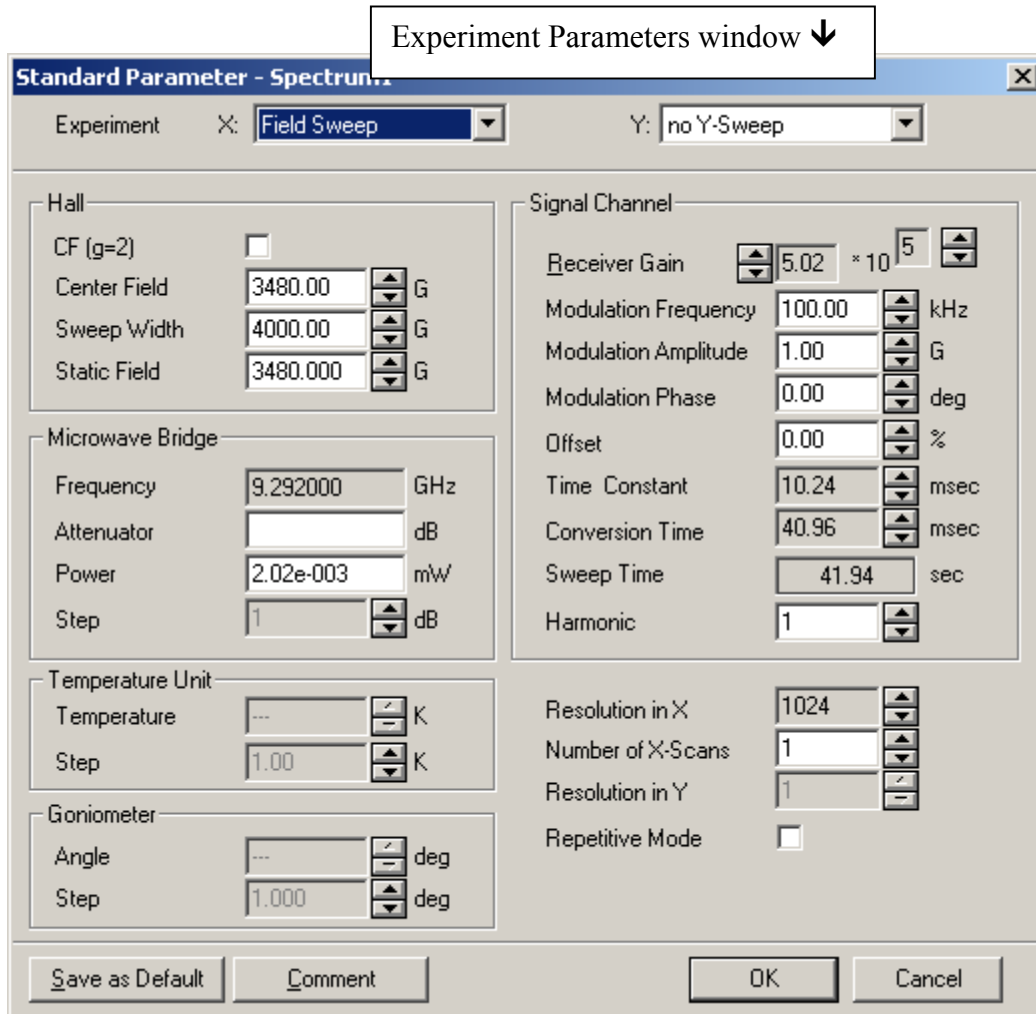
When you click on the New icon on the far left of the toolbar, you will get a new spectrum window with the instrument acquisition parameters set to their default values. These are the parameters that we use for collecting blank scans both before and after using the EPR to check for cavity contamination.

You will probably need to change some of these parameters to suit your experiment. Do this by clicking on the Experiment Parameters button on the toolbar. The parameters you need to be concerned with are the following:

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 collect a spectrum into a window, it is stored only in the computer's volatile RAM memory. To save it permanently on disk, you must select the window, and do a File – Save As. If you want to save the file in ASCII format, do not choose ASCII under the save as file type. There is a flaw in the software, and doing this will destroy your data. ASCII conversion must be done instead in the Win-EPR data manipulation program, and not in the Win-EPR data acquisition program.

V. 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) Exit from the Win-EPR acquisition software. This will log you off.
- 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.