EMX-EPR Spectrometer Guidelines

1. Choose Win EPR on desktop.
2. Open Microwave Bridge Control (MW in toolbar).
4. Check that the Center Field is close to 500 G.
5. Turn on power supply.
6. Increase center field value by 500 G increments until approximately 3500 G. Watch the “Halt Field” – wait for it to turn green before adding next increment.
7. On MWB, select “Tune.” Lower attenuation to 40 and adjust Frequency until the dip is in the middle. Then adjust Signal Phase so the dip is sharp and the line is flat on either side of the dip. Put attn. Back to 50-60.
8. Select Operate. Tune AFC and Diode at attenuations from 60-10. At attenuations 60 and 50, adjust Frequency and Bias; 40,30,20 adjust Frequency and Iris.
9. At Attenuation 10, use signal phase to move diode as far to the right as possible. Then use the Iris to move the diode meter back to the middle of the green. Continue to adjust AFC and Diode at different attenuations until the field is stable at all powers.
10. Open Spectrum window and check standard parameters. Enter the Center Field and Static Field values according to the frequency and the little chart taped to the monitor. These values should be the same and should correspond to the frequency value obtained after tuning the magnet.
11. Run spectra.
12. At the end of your experiment, return to the MWB. Increase the attenuation to 60, and go from Operate to Tune to Standby.
13. Reduce the Center field in increments of 500 G. Stop at 500 G. Turn off power supply.

NOTE: This list of instructions is not a replacement for training! Please see Tracy if you have any questions or would like training.

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Current List of Approved Users:
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Running the Standard for the EMX-EPR Spectrometer

If the instrument is behaving abnormally, please try running a standard sample to help discern what the problem is. The standard is TEMPO.

![TEMPO](image)

TemPO
1x10^-4 M in hexanes
Ar sat'd for 5 minutes in a pipette tube
stoppered with a rubber septum

A standard solution is stored in the fridge in 750A. A new solution is made up each month.

To run the standard:

1. Use a pipette tube that is sealed at the bottom.
2. Add enough solution so that the tube is half full.
3. Argon saturate the solution for 10 minutes with slow bubbles for ~5 minutes, using a 20-gauge needle.
4. Stopper the tube with a rubber septum and run the standard. The parameters, including the modulation amplitude, time constant, microwave power, etc. are listed on the spectrum of TEMPO. This spectrum is included below and is also pasted on the inside back cover of the EMX-EPR log book.
5. Save the spectrum that you obtain in Data/Standards. The filename you should use is TEMPO_the date. For example, a standard spectrum run on Oct 2, 2002, should be TEMPO_1002002.
6. Print out the spectrum (at 40% of an 8.5x11” page) and paste it directly into the log book.