Guide to Operation of the Bruker E500 EPR Spectrometer

(draft)

General information: The Bruker ElexSys E500 EPR spectrometer is operating at the X-band frequency in the CW mode. There are two resonators available to use. The first one is a super high sensitivity resonator with optical port, suitable for most organic and inorganic samples. The second one is a dual-mode ER4116DM resonator, which allows the microwave being applied either perpendicular or parallel to the sample axis, and is specifically designed for anisotropic studies of triplets, biradicals, and of transition metal and rare earth ions containing "forbidden" fine structures or hyperfine structure transitions.

Temperature control of the sample can be done in three different ways. A variable temperature system using liquid nitrogen boil-off air is regularly set up for temperatures ranging from room temperature down to about 100K. A finger Dewar can be used for 77K, and a liquid helium cryostat system can be used for 4K.

Start-up Procedure: (usually you can ignore 1-5)

1. Turn on the chilled water by turning the yellow valve handles from horizontal position to vertical position.
2. Turn on the nitrogen air by turning the red valve to horizontal position.
3. Turn on the heat exchanger unit by flipping the power switch to 1 (on) position.
4. Log on to your group account and start the Xepr program by double-clicking the Xepr icon.
5. Turn on the spectrometer by pressing the green button on the left-hand side of the front panel
6. **Turn on the magnet power supply by pressing the green POWER ON button**.

Sample loading:

1. Loosen the collet by unscrewing the nut until the opening is large enough to insert your sample tube.
2. Figure out how far you need to insert the sample tube by measuring the length of the tube from the bottom of the cavity to the top of the collet sample holder.
3. Insert your sample carefully into the cavity.
4. Tighten the nut to secure the tube at the proper position.

   (If you use the finger Dewar, make sure it is clean inside to avoid too much bubbling of liquid nitrogen. Clean your tube before putting it into the Dewar. The sample tube should be 9 inches or longer for this operation.)

Cavity tuning procedure:

1. Connect the spectrometer by using the pull-down menu to select Acquisition – Connect To Spectrometer and click the OK button (the Server name should be localhost). *This step can be*

ignored if the spectrometer is already connected, which is indicated by the presence of three meters and other icons shown on the bottom of the program.

2. In the Xepr program window click on the icon with the tuning curve (downward peak), which will open the tuning window. You can drag and move this window to the second screen on the right.
3. Click on the Tune button to show the tuning curve (it will take a few seconds to show this).
4. Click on the Auto Tuning Up or Down button to start autotuning. If successful, both the Diode Current and the Lock Offset will be centered, and Operate, Leveled, and Calibrated buttons will turn to green color. You can then close the tuning window, or just leave it there.

Starting the experiment:

1. If you have saved a data set previously, you can load it up from the disk and save it in the memory and start with the same parameters by clicking on the Create Experiment from the Dataset button. Otherwise, click the Exp button to create a new one.
2. Click on the icon marked with 23 to open the parameter window, where you can make any adjustment to change the center field, sweep width, microwave power, modulation frequency, modulation amplitude, receiver gain, conversion time, and time constant, etc. Typical values are as follows:
    a. Center field: 3400 G
    b. Sweep width: 50-100 G for organic radicals, 3000G for transition metals
    c. Microwave power: 1mW to 20 mW (higher for wider sweep width)
    d. Modulation frequency: 100 KHz
    e. Modulation field: 0.1 to 10G (depending on the linewidth)
    f. Receiver gain: 1x10**4
    g. Conversion time: 60-200 ms
    h. Time constant: match with the conversion time (higher value filters out more noises)
3. Start the experiment by clicking on the Play button (Clicking on this button again aborts the experiment).
4. Use the pull-down menu, select File – Print Viewport to open the printing window and select options to print the spectrum.
5. Save the data by using the pull-down menu, selecting File – Save and enter filename, title, and path, then clicking the Save button.

Shutdown procedure: (you need to do only 1, 3, and 7)

1. **Open the Tune window and click the Standby button, then close this window.**
2. Disconnect the spectrometer from the Xepr program by using the pull-down menu, selecting Acquisition – Disconnect From Spectrometer.
3. **Turn off the magnet power supply by pressing the POWER OFF button**
4. Turn off the heat exchanger by flipping the power switch down.
5. Turn off the chilled water and the nitrogen air.
6. Turn off the spectrometer console by pressing the red button.
7. **Remove your sample and cover the cavity with a piece of Kimwipes or a cup.**