**EMX plus EPR general SOP**

10/12/2017

***NOTE:*** *If you want to do cooled experiments you may need to start up parts of the instrument* ***24 hours*** *before the planned use; on the day of the planned use and before you can start your experiments it will take* ***1 hour*** *to purge the system with He, and an additional ±* ***2 hours*** *to get down to 8K, so please plan accordingly*

**Starting up the instrument for room Temperature experiments**

1. Turn on main cooling:
2. Open both water valves on wall
3. Turn on power to Haskris heat exchanger (only the one closer to the entrance)

***NOTE:*** *If the temperature does not start to go down immediately, turn off power to heat exchanger and reset compressor by pressing the black button behind the front panel.*

1. Start up the instrument:
2. Press black button in lower right of the EMXplus console (behind lower panel)
3. Press circular button on front of the console
4. The EMXpremiumX bridge will come up by itself, wait until the blue light stops blinking
5. Start the Linux PC:
6. Log in with username “xuser” and password “user@xepr”
7. Launch the Xenon software

**Starting up the instrument for cooled experiments (≥8K)**

1. Turn on two pumps **24 hours** before planned use, if they are not running already (should be running continuously except for servicing):
2. Switch on Pfeiffer HiCube, watch pressure go down to 4x10-3 (pressure 340, speed 309)
3. Switch on ROC-R purge pump
4. On the day of planned use, set up He and dry N purge:
5. Open up the Helium tank and set He regulator to 100 psi
6. Open up valve marked SUPPLY going to cold head, check flow meter in front of bridge to make sure He is actually flowing, purge for **1 hour** to avoid condensation inside the cold head
7. Open up the Nitrogen tank and set N regulator the 5 psi, regulate down the flow until you can barely feel flow coming through the slotted window in the front of the resonator; this keeps the sample from freezing in
8. Turn on main cooling:
9. Open both water valves on wall
10. Turn on power to both Haskris heat exchangers

***NOTE:*** *If the temperature does not start to go down immediately, turn off power to heat exchanger and reset compressor by pressing the black button behind the front panel.*

1. Start up the instrument:
2. Press black button in lower right of the EMXplus console (behind lower panel)
3. Press circular button on front of the console
4. The EMXpremiumX bridge will come up by itself, wait until the blue light stops blinking
5. Turn on the Mercury iTC temperature controller
6. Start the Linux PC:
7. Log in with username “xuser” and password “user@xepr”
8. Launch the Xenon software
9. After **1 hour** of He purge, and once the second Haskris heat exchanger has reached **60°**, turn on the ColdEdge compressor; cooling down to 8K will take about **2 hours**

**Shutting down the instrument**

1. Close the Xenon software:
2. Open the TUNING panel and set the Microwave bridge to “Stand By”
3. Exit the Xenon program
4. Remove your sample from the resonator and cover the collet with the black plug
5. Shut down the instrument:
6. If cooling was on, turn off the ColdEdge compressor and the Mercury iTC temperature controller
7. Close the He tank and the SUPPLY valve going to cold head
8. Press circular button on front of the EMXplus console
9. Press black button in lower right of the console (behind lower panel)
10. Turn off power to Haskris heat exchangers
11. Close both water valves on wall

***NOTE:*** *The Pfeiffer Hi-Cube and the ROC-R purge pump should be kept up and running continuously. Only turn them off for servicing.*

**Running a sample or experiment**

1. Loading your sample into the resonator:
2. Turn the black knob on the He controller in front of the bridge so the gauge goes to max; this will create positive pressure inside the resonator for easy sample exchange
3. Wipe off the outside of your sample tube to avoid contaminants in the resonator that would permanently disturb the EPR signals
4. Loosen the top collet nut, remove the collet plug and insert your sample tube as straight as possible; gently and slowly push it in by holding it close to the collet nut; stop once you feel resistance! ***Do not force tube further in***! Gently tighten the collet nut.
5. If using cold head, set desired temperature on the Mercury iTC:
6. Push the CONTROL button, select SAMPLE in upper box, put in desired temperature, change from AUTO to MANUAL, close

***NOTE:*** *The displayed sample temperature is not exactly the same as the temperature inside the sample since the temperature probe is located 2cm below the sample center and the temperature shows some He pressure dependency; refer to the TP dependence charts (on argenta2 server)*

1. Tune the bridge and the resonator:
2. In the Xenon software, switch to ACQUISITION MODE and open the TUNING panel
3. Set the Attenuation to 30 dB
4. Press either UP or DOWN to start the Autotune; this will adjust the frequency, phase, and bias of the bridge and the matching of the resonator; if an error occurs, or if your sample strongly absorbs microwaves (like H2O), tune manually (see below)
5. Wait until the TUNE / UNLEVELED / Q-VALUE / FIELD status sections turn green
6. Acquiring a spectrum:
7. In the Control Panel for Spectrometer (bottom of window), choose Acquisition type (1D\_FieldSweep for simple field sweep experiment)
8. Click the SAMPLE INFO button ( **i** ) and enter info
9. Click the ORGANIC RADICALS or TRANSITION METALS default parameter button (bottom of Field Sweep panel)
10. Acquire a spectrum by pushing the RUN button ( **>** )
11. Adjust MW POWER / ATTENUATION / GAIN in the field sweep panel (left), hit RUN and adjust again until spectrum appears optimal
12. Center the spectrum and adjust the sweep width by pushing the SWEEP TOOL button (**<->**) and then clicking and dragging the center field marker to the center of your spectrum; click and drag the ends of the sweep tool to adjust sweep width
13. Remove the current active tool by using the REMOVE button (trash can in top bar)
14. RUN again and acquire a centered spectrum
15. Saving your data:
16. Click the SAVE TO DISK button (top left), choose the directory, and save

**Manually tuning the bridge and the resonator**

1. Open the Tuning panel
2. Click **Tune**
3. Set the Attenuation to 30 dB
4. Turn the reference arm off
5. Use the Frequency slider to find the dip
6. Turn the reference arm on
7. Adjust the Phase slider for maximum dip height and symmetry of the dip
8. Click **Operate**
9. Adjust the Frequency slider until the Lock Offset [%] is around zero
10. Change the attenuation to 50 dB
11. Adjust the Bias slider until the Diode Current is around 200, click the reference arm on and off a couple of times to eliminate hysteresis and continue to adjust the Bias slider until the Diode current is around 200
12. Set the attenuator to 40 dB
13. Adjust the iris until the diode current is at 200
14. Set the attenuator to 30 dB
15. Adjust the iris until the diode current is at 200
16. Set the attenuator to 20 dB
17. Adjust the iris until the diode current is at 200
18. Set the attenuator to 10 dB
19. Adjust the iris until the diode current is at 200
20. Adjust the Phase slider to find the local maximum of the Diode current
21. Adjust the bias, if needed, to bring the Diode current back to 200
22. The system should now be tuned. Close the tuning panel window and run your sample.