

# Notre Dame EPR User Checklist

Bruker EMX EPR Spectrometer, X-band, 9.75 GHz

10/21/2021

**Training:** All new users must be trained by NMR center staff before they can use the spectrometer. Email your training request to Evgenii Kovrigin at [ekovrigu@nd.edu](mailto:ekovrigu@nd.edu). This checklist and training tutorials are found on:

<http://nmr-center.nmrsoft.com/EPR/EPR.html#RTepr>

**Location:** Stepan Chemistry Hall, room 162

**Sample Tubes:** 4 mm **DO NOT use 3 or 5 mm tubes!**

## Samples:

- crystalline or non-polar solutions may be loaded directly in 4 mm tubes. The height of the sample in a tube should be less than 2-3 cm (excess height sits outside of the resonator).
- **polar** solvents should be put in a **capillary** (10-20 $\mu$ L), which is then put in a 4 mm tube. If polar solvent is loaded directly in a 4 mm tube, it will be impossible to tune.

**CRITICAL: See water on a floor? DO NOT ENTER! Notify NMR staff immediately.**

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## Startup sequence

1. Open water supply valves on the wall (handles - to vertical position).
2. Turn on the Haskris heat exchanger.

**Before you proceed, check that you do not see any water leaks!**

3. Flip UP the switch on the **RIGHT** breaker box on the wall. (Left box is for a cryogenic operation)
4. Turn on the EPR power supply by pushing the leftmost button.
5. Once the red LEDs have lit up and turned dark again, push the "ON" button on the power supply.
6. Turn on power to EPR spectrometer (on the back of the spectrometer cabinet).
7. Wait 2-3 min.
8. Open the Xenon software.
9. Click Acquisition > Connect to Spectrometer.

## Inserting the sample

1. Switch tune interface to "Stand by" mode
2. Remove the cover cap from the dewar tube (pull up)

3. **GENTLY** put your sample tube into the dewar opening and **SLOWLY** lower it until it touches quartz bottom of the dewar.
4. Before changing a sample: switch the tune interface to "Stand by" mode.

## Saving your data

- You should save your data to **/home/xuser/data/your-user-name**. NMR Data Server pulls data from this folder every few minutes.
- **IMPORTANT NOTE:** The folder, which is open in Xenon by default, is **not uploaded** to the NMR Data Server!
- To download data on your computer, go to **nmr.nd.edu, Data Server, NMR Data Server v5.0**, and click on "**EPR**" in the list of names.

## Power down sequence

1. Go to Acquisition > Disconnect from spectrometer. DO NOT "X" directly out of the program before disconnecting.
2. Remove your sample
3. Close dewar opening with the cap.
4. Turn off power to spectrometer (on back wall of the spectrometer cabinet)
5. Turn off the power supply by pushing the red button, followed by the left-most power button.
6. Turn off the switch on the **RIGHT** box on the wall (flip DOWN).
7. Turn off the Haskris heat exchanger.
8. Close the chilled water valves on the wall (handles - to horizontal position).
9. **Check for water around all equipment.**

## Troubleshooting

Problem: My sample is in polar solvent and does not tune

**NOTE: This is expected!**

If your sample is polar and fills entire volume of a resonator (about 2 cm) it will absorb microwaves too much and will prevent tuning of the resonator.

**Examples:** water or DMF in a 4 mm tube.

**Solution 1:** Use a capillary with 10-20  $\mu\text{L}$  of your sample inside an empty 4 mm tube.

**Solution 2:** insert your 4 mm tube through a sample well cap and adjust height for partial insertion in a cavity:

- adjust tube height so the sample will still be outside of a resonator when inserted;
- Turn on tuning and tune the empty cavity;
- Switch tuning screen to Tune : Stop mode
- Start pushing the tube down very gently while observing the tuning dip
- When it is 1/3 or 1/2 between fully out and fully in, try to tune again.

Problem: Tuning stops with "Iris lower limit"

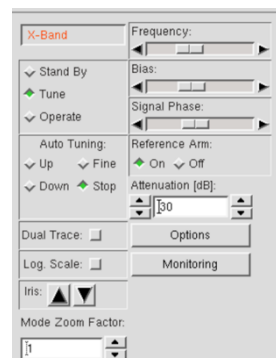
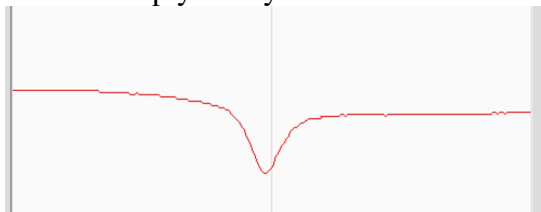
This problem may occur if the solvent is too polar (see above) or if the resonance condition occurs too close to the edge of the Iris Motor range.

Solution to this situation is to tune manually (which you can do in other cases too).

**To tune manually**, in the **Microwave Bridge Tuning** screen:

1. Turn on tuning mode: click **Tune**
2. Click **Stop** in **Auto Tuning** section (if it is not clicked)
3. Click **Options** button and reactivate the iris motor by clicking off and back on the **Iris Motor Present**.
4. Shift **Bias** slider all the way to the left
5. Find the frequency of the tuning dip: move the dip to the center line using **Frequency** slider then click arrows for small steps.

NOTE: Empty cavity tunes at around 9.45 GHz



6. Find **Iris** position for the deepest dip: click and hold arrow up or down buttons. If autotuning previously stopped with a message "Iris Lower Limit", click Up, and vice versa. Click **Attenuation** arrows up or down to make sure the tuning dip fits in the screen (arrows on the right make 1 dB steps).
7. Move **Bias** slider to the middle range
8. Move the **Signal Phase** slider so the tuning dip has a negative absorptive shape
9. Click on **Operate** in Tune section
10. Click on **Monitoring** button to see big gauges
11. Shift **Bias** (click arrows) to adjust it to 200

12. Shift **Lock** to 0 using **Frequency** arrows.

NOTE: Lock is **VERY** sensitive. One arrow click is about 20 units on a lock gauge (1/10 of full range).

13. All indicator windows on a status bar must be green at this time indicating a tuned state:



Problem: Iris motor shaft disconnected from the Iris screw

**Ask NMR staff to show you how to do this adjustment!**

If the retaining ring of the iris screw got detached, the iris motor shaft may jump out of the screw.

To fix it:

1. Go to Tune panel, click Stand by
2. Click Options button, choose Iris Run Down checkbox  
If it does not respond, check on/off the Iris Motor Present checkbox
3. When a question box pop up DON'T CLICK.
4. Go to the instrument sample holder and take the tube out.
5. Unscrew the retaining ring of the Iris  
Use two hands with one middle finger of each to rotate the retaining ring
6. Pull the shaft out
7. Click on Yes button in the "Please decide" box  
Motor will begin rotation to reach the lower limit position
8. Once the motor stops, use a "ruler-screwdriver" to turn the tuning screw CW (half turn each time). If your tube is inserted, take it out or pay attention not to break it with your screw driver.
9. Finish rotating the tuning screw to align it with the end of the motor shaft
10. Put the shaft in and secure with a retaining ring (CCW, two hands needed).

## Contacts

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