EPR Checklists

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These checklists are intended to supplement not replace proper training by Justin Douglas. Please use these checklists to avoid costly mistakes and maximize your research productivity.

TURNING ON THE INSTRUMENT

Breakers on

Haskris on (wait until water temperature = 65 °F)

Power supply on (switch on back)

Console and bridge on (wait for bridge to stabilize)

Log into workstation and open Xenon.

Open microwave bridge tuning window and switch to tune mode. Let the spectrometer warm up ~1 hour

Turn on magnet prior to recording a spectrum.

COOLING THE CRYOSTAT (THIS IS A TWO-PERSON JOB)

Is the cryostat pumped down to <10-6 mbar? Is the Teflon ring on the transfer line?

Purge transfer line with N_2 gas for ~5 minutes (mandatory for liquid helium, optional for liquid nitrogen)

Insert transfer line into dewar.

Check flow at bayonet.

Insert bayonet into cryostat, but DO NOT engage nut. Turn on pump.

Wait ~10 minutes (Sound will change). Engage nut.

Make sure nitrogen purge gas is flowing.

Turn on ITC. Set needle valve to ~1/4 to 1/3 of a turn open. Set PID and heater to auto and set temperature to desired value.

LOADING/UNLOADING SAMPLE WHEN THE CRYOSTAT IS COLD

Confirm that the bridge is in tune mode

Turn heat to manual and heater output to 0%

Turn off pump. For liquid helium, the pressure will rise and helium flow will drop to zero, then rise to ~0.5 l/hr. These changes are less obvious for liquid nitrogen. Be patient.

Pull top of chimney stack straight up.

Put chimney stack with EPR tube or placeholder back onto top of cavity

Turn on pump

Turn heater to auto and set temperature to desired value.

LOADING/UNLOADING SAMPLE WHEN THE CRYOSTAT IS WARM

Confirm that the bridge is in tune mode

Loosen collet or brass fitting.

Pull sample straight up out of the cavity

Insert new sample straight down into cavity

Tighten collet or brass fitting.

TUNE CAVITY

Open microwave bridge tuning dialog box

MAKE SURE CAVITY IS CORRECT!

Switch to tune mode

Set attenuation to 20 dB and center on appropriate dip. Adjust phase so that dip is symmetric

Set attenuation to 50 dB. Set to operate mode. Adjust bias so that diode current ~200 μA. Don't touch bias again

Set attenuation to 40 dB. Click fine tune button.

Set attenuation to 30 dB. Click fine tune button

Set attenuation to 20 dB. Adjust phase to maximize diode current. Click fine tune button.

Set attenuation to 10 dB. Click fine tune button.

Check for 50-10 dB. Make sure diode current ~200 μA and lock offset ~0%.

Set attenuation to 33 dB and switch to tune mode to measure Q-value.

Switch operate mode. Set attenuation to 30 dB. Click fine tune button.

QUICK SPECTRA ACQUISITION

Open microwave bridge tuning dialog box and MAKE SURE CAVITY IS CORRECT!

Set acquisition parameters by clicking “Organic Radical” or “Transition Metal” button on left side near bottom.

Is your modulation amplitude too high for your cavity? Don’t overmodulate the cavity or you could damage the probehead. Though sometimes unavoidable, try to work at 4G or lower.

Click Play button to acquire spectrum.

Save data to disk. Be sure to save in /home/data/username folder. Be sure to give both a file name and a title.

I CAN’T SEE A SIGNAL WHAT DO I DO?

Set power to 10 mW, set modulation amplitude to 1 G and run a fast scan with a short time constant. Do you see a signal now? Check instrument performance on a sample of TEMPOL or some other positive control. Think about the concentration of your sample and the temperature of the cavity.

QUICK PARAMETER OPTIMIZATION

You will need some idea of what the width of narrowest signal is.

Is the signal saturated?

> Reduce attenuation by 6 dB and record spectra again. Is this spectrum 1/2 as intense as the 1st spectrum? Yes – the first spectrum isn’t saturated. No – the first spectrum is saturated. In general, you want to use the highest power (lowest attenuation) possible without saturating.

Are you overmodulating your signal?

Reduce modulation amplitude by 1 G and record again. If line widths are narrower in the second spectrum, then modulation amplitude was too high for the first spectrum. (Note: unless you are trying to measure very small hyperfine it is usually acceptable to have the modulation amplitude ~ 1.5 times the natural line width.)

Is the spectral width wide enough to see all features in your spectrum?

If you are not sure, then be conservative and record again with larger spectral width. If possible the distance from starting point to 1st signal should be 10 times the width of the smallest signal.

Is the time constant set correctly?

Your time constant should be equal to or less than the conversion time.

Am I collecting the right number of points?

The magnet field step size should be smaller than 1/10 of the narrowest linewidth. Collecting too many points just takes up more room on the disk, which (for now) isn't a problem. Remember you adjust number of points by changing the pts/mod. amp.

WARMING CRYOSTAT (TWO PERSON JOB)

Wear gloves and goggles!

Remove sample and put chimney stack top with place holder on top of cavity.

Turn heater to manual and heater power to 0%.

Close needle valve.

Wait until T > 200K.

Turn off purge gas.

Turn off pump and ITC.

Disengage nut coupling transfer line to cryostat and remove bayonet.

Remove transfer line from dewar.

Put "bunsen valve" into the end of transfer tube arm.

Seal dewar.

SHUTTING DOWN SPECTROMETER

Make sure spectrometer is in standby mode

Close xenon and log out of workstation

Turn off magnet, bridge, console and power supply

Wait 5 minutes

Turn off Haskris.

Turn off breakers