

Order of Operations for First Time Solids Spectrometer Use

(1) Grab the probe you wish to use from the probe cabinet and ensure that the correct top plate is installed. This step can be skipped if your probe was in use by the previous user.

(2) Use the Varian probe manual to platform the probe for the nuclei you are interested in.

(3) Use the sweep meter to roughly tune the X and Y channel of the probe on the bench.

(4) Pack your sample into the appropriate rotor using the rotor packing instructions.

(5) Fill out the rotor usage log.

(6) Load the probe into the magnet and connect all of the cables.

(7) If you have changed the probe you must execute an 'edhead' command and select the appropriate probe. You must then load the latest shim files for that probe. These can be found by typing 'rsh' into the command line and selecting the appropriate shim file for your probe and temperature.

(8) Open up your previous reference data sets and use the 'edc' command to create new data sets for referencing with the correct dates. This is usually the adamantane and hexafluorobenzene data sets.

(9) Load the adamantane rotor into the probe and spin the sample to a speed of 8 kHz.

(10) Open up the reference data sets and ensure that all of the nuclei you are interested in are listed in the routing. This can be checked by typing 'edasp' into the command line. Once all of the nuclei are listed and correct you can type 'wobb' and go and tune the probe.

NOTE: You must ensure that **ALL** of the channels are tuned correctly before moving on to the next step!

(11) Look in the probe log book and use the previous user's **WEAKEST** proton power to quickly acquire a 4 scan proton spectrum for adamantane. Process and properly phase this spectrum. Set the RF pulse frequency (o1p) on resonance with the proton peak. Quickly re-acquire this spectrum and make sure that the phase and processing are still good.

(12) Use the 'popt' routine to measure the 90 degree pulse width and power for proton. Be sure that these powers do not exceed the pulse width specifications of the probe and remember that you **MUST** record these values in the appropriate probe log book. Also, remember that you **MUST** measure all of the powers for each of the pulse widths listed in the probe log book.

(13) Now that you have measured the powers you can reference the chemical shift of the standard. This is done by simply clicking on "calibrate axis" in the processing menu or typing '.cal' in the command line. Be sure to write down your SR value as this can be useful later on.

(14) Repeat steps 11-13 for carbon. **BE SURE** that the power used for proton decoupling (plw12) is **NO MORE THAN** the power used to reach half of maximum pulse width. For example, if the maximum pulse width was 2.5 μ s, you should use **NO MORE THAN** the power used to reach a 5 μ s pulse width.

(15) Check the carbon line width at half height for adamantane. If the line width is less than 12 Hz your shim settings are ok, otherwise you must contact the manager and ask for assistance in shimming the magnet.

(12) Repeat steps 11-13 for fluorine using hexafluorobenzene as a standard. This step can be skipped if you are not working with a fluorine containing material.

(13) Repeat steps 11-13 for the fourth nuclei and be sure to use the appropriate reference standard. This step can be skipped if you are only working with a triple resonance experiment.

(14) Now you are ready to begin carrying out measurements on your sample. The following are some key steps you can take to make sure your experimental life is much easier:

(i) The first thing that should always be done on an unknown sample is to find the appropriate recycle delay (d_1) to use. This is done via a T1 saturation recovery experiment.

(ii) When acquiring the spectra you will want to set the transmitter offset (o1p) beside the main resonance line in your spectra (*i.e.* not directly on the line). This makes detection of the FID for low gamma nuclei, such as ^{13}C , much easier.

(iii) Make sure that you write down the offset for each nucleus in your sample (o1p) as this is important when setting up decoupling.

(iv) Use a **SEPERATE** experiment number for each nucleus and experiment type. Be Sure to use a good title that clearly labels what each experiment number contains.

(v) Be sure that your experiments are referenced correctly. This can be done by checking the SR value you wrote down in step 13 above.

(vi) DO NOT EXCEED the 90 degree powers you measured earlier as this can lead to probe arcing and very expensive probe repairs/replacements.

(vii) If you are setting up a CP experiments you must be careful to not set the powers during the spin lock too high. Setting this power too high can cause probe arcs and creates decoupling issues in the sequence. In most cases you will want to set the ^{13}C power to 70 percent of the power used to meet the probe specifications.

(ix) Always remember that the decoupling power is given by the parameter plw12 or plw13.

NOW YOUR ON YOUR WAY TO ACQUIRING THE SPECTRA YOU NEED!