

## Observing new nuclei on the NMR

### *Set up configuration*

rpar ?? creation/copying of CORTAB file?/

edhead            add new nucleus, then click edit probe parameters, followed by peak power parameters (set peak power to 0, for reference:  $^{13}\text{C}$  is at -3.00).

-Define as current probe, click seen and exit.

-Close pop-up “edit spectrometer parameter” window.

edprosol            Select the desired nucleus, enter dummy Pulse Width and Attenuation Level values into both Observe and Decouple. Use values for a nucleus with a similar gyromagnetic ratio. (15  $\mu\text{s}$  should be a good starting point)

-Copy to probe, save to all solvents. Save and quit

getprosol

atmm                manual tuning and matching required for first time

lock <solvent>

topshim            shim sample

### *Optimize parameters*

Acquire spectrum, expand around peak. Click the red lightning bolt arrow icon in the upper right of the screen to set the offset of the peak. Select O1.

Acquire a spectrum again: you should get a peak. Right click and select the ‘save display region to’ option. Select Parameters F1/2 and click OK.

ns                    Set the number of scans for each experiment (a small number here is good eg, 4) and DS=0

try POP rather than PAROPT?

paropt              Here the parameter being optimized should be P1 (=90° pulse-length).

-Start at the dummy P1 value that was originally entered in. Select the size of the increments and the number of experiments to cover a given range around the initial value.

-If FID only gets worse, try going in the other direction by using a negative increment. The range can also be changed.

Watch the intensity of each of the FIDs when they begin. Keep track of the P1 value that is currently running. The optimum P1 value corresponds to the FID with the largest initial intensity.

An error will most likely come up at the end, disregard it.

When the experiment finishes, set the P1.

To obtain a more accurate value, try P1 values roughly 4x this value; look for the null. (Divide by 4 to get the actual P1 (PW90) value.

edpsol            set pulse sets in Observe to optimum P1 value.

Save, copy to probe, save to all solvents.