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CHM 398: NMR Topics
Independent Project

Observing new nuclei on the NMR

Set up configuration

rpar *or* eda	recall parameter set if one exists (rpar). If not, read in a similar set. edit the acquisition paramters (eda) to set F1 to the desired nucleus. Save as a checkfile (wpar).
edhead	add new nucleus, then click edit probe parameters, followed by peak power parameters. Set the peak power to a value similar to a nucleus with a nearby frequency.
	- Define as current probe, click seen and exit.
	- Close pop-up edit spectrometer parameter window.
edprosol	Select the desired nucleus (righthand column), enter a dummy p1 value (15 μs should be a good starting point) into both F1 and F2. Enter a power level (PL1) similar to the nearby nucleus. (for reference: ^{13}C = -3.0)
	- Copy to probe, save to all solvents. Save and quit
getprosol	
lock <solvent>	
topshim	shim sample
atmm	manual tuning and matching required for first time

Optimize 90° Pulse

	Acquire spectrum, expand around a single peak. Click the red lightning bolt arrow icon in the upper right of the screen (or type dpl) to set the spectral window to the expansion. Select 01.
	Acquire a spectrum again: should get 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: NS = 4, DS = 0. Also verify that the recycle delay is relatively short — D1 < 5 sec)
popd	Use popd to optimize the P1. (paropt is simpler, but popd behaves better).
	- set the parameter to optimize to P1 (the 90° pulse-length). - set the optimization to "posmax" (max value of a positive peak). - enter starting and ending values, and # experiments. - click "start optimiation".

An approximate P1 value will be returned. To refine the value, multiply it by 4 and search for a zero-crossing around the quadrupled value (= 360° pulse).

When the experiment finishes, set the P1 to ¼ of the zero-crossing value.

edprosol

Double-check that parameters being changed are for the correct nucleus (right column).
Set pulse sets in F1 to optimum P1 value.
Save, copy to probe, save to all solvents.