SPT (Spin Tickling) and NOE

Selective Population Transfers

Selective Population Transfers are best done as SPI (Selective Population Inversion), which requires a selective 180° pulse to be created for the specific resonance. A less effective, but more convenient method is to presaturate the resonance by CW decoupling. Thus, instead of being inverted, the population is equalized: halfway to inversion! This is preformed very much like selective decoupling, except that the decoupler is off during the acquisition.

Homonuclear NOEs may similarly be generated and the differences obtained manually. Bruker has several convenient parameter sets which allow irradiation at a list of frequencies, (and also acquire a reference spectrum); this is much more convenient than obtaining the NOE difference spectra one frequency at a time.

Reference Spectrum

1. Insert a sample, lock, shim, and record a normal ^{1}H spectrum.
2. Note the position (in Hz or ppm) you wish to decouple. For SPT, pick a single line of a multiplet; for NOE, pick the center of the multiplet. This is easiest done by placing the cursor on the peak, and recording the peak position at the left of the screen.

Irradiated Spectrum

3. Create a new dataset in a new window.
   The easiest method seems to be reopening the reference spectrum from step 1 in a new window, then creating a new EXPNO within the folder (edc).
4. Recall the proton presaturation parameters: "rpar NOEDIFF all". "getprosol" as usual — this is important, as several important parameters are not defined in the NoeDiff set, but are imported during the getprosol.
   Take a look at the pulse program (⎍ button under Pulse Program tab), noting the timing of the decoupling.
5. Several parameters need to be set:
   - Note the P1 value (90° pulse length). Change it to ⅓ of its value (30° pulse; typically about 5 μs).
   - Set the decoupler frequency offset, O2, to the value you obtained in step 2 above. (O2P if your value was in PPM).
   - Adjust the decoupler power level, PLdB14: it should be around 70 dB for SPT, but lower (more power/wider envelope) for NOE.
8. Acquire the spectrum as usual (RGA; ZG; EFP).
9. If necessary, tweak the decoupler power and repeat from step 7.

IMPORTANT: *the PLdBxx settings are attenuation settings*. Higher numbers give lower power.
Although there should be safety-checks in the software, be careful to never send too much power through the coils — high power decoupling (small settings) can fry them. ($$$$)

Multiple Display and Difference Spectra

10. Return to the Reference Spectrum and set the LB value for it to match the Irradiated Spectrum's. Transform the spectrum (efp). A larger LB value will allow for better subtraction in the difference if the peaks do not exactly coincide.

11. Enter multiple display mode. ("mdisp" or ⊥⊥ button in top row).
    It is important that the reference be the first spectrum in the stack: the order of the others can be changed, but the first one in cannot be moved.
12. Open the other spectra you wish to display by left-dragging them from the Browser window (on the left of the screen) into the main window.

    If necessary, you can toggle between stacked and superimposed spectra with the ⊥⊥↕ button.
13. Obtain the difference spectrum.
    Select a spectrum by clicking the box at the right of the screen.
    The △-button will generate the difference between the selected spectrum and (the sum of all ?) the other(s). (The ∑-button will similarly display the sum of the spectra.) You may need to adjust the vertical scale (⬍S) of one spectrum or shift it (⟷S) to get peaks to cancel reasonably.