1D NOESY

Experiment name on 500 ---- dc_1D_NOE

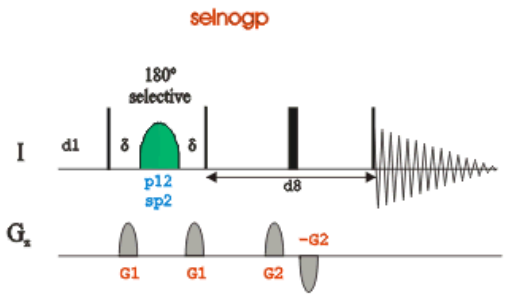
Revision 150518

Introduction

The 1D NOESY is a quick and specific option when the completeness of a 2D NOESY isn't necessary. Instead of revealing all the possible Nuclear Overhauser Effects as a 2D does, the 1D irradiates only a particular resonance. Any protons that experience an NOE from that resonance should be identifiable if properly set up.

The relevant parameters for an optimum 1D NOESY are:
01p – the new center (in ppm) where the resonance of interest will be irradiated. (Be sure your SW is wide enough.)
p12 - duration of the selective 180 pulse (between 10-80msec)
d8 – mixing time (0.3-0.5sec typically for small molecules)

The pulse program is selnogp



Source: Bruker 1D Proton selective experiment tutorials

Considerations:

- The best option is to use the shorter selective 180º pulse that achieves clean excitation of the selected resonance without disturbing neighboring resonances. In some cases, the use of long selective pulses can undergo important sensitivity losses due to relaxation effects.
- If critical peaks to be irradiated are very close (<30 Hz) to other peaks, then the selectivity of the 1D version will not be sufficient and the 2D version will be needed.
- Dissolved oxygen or other paramagnetic species such as Cu2+ can reduce or completely quench the NOE. For small molecules, it is extremely important to remove dissolved oxygen. For large molecules, the removal of oxygen is not critical. Removal of oxygen must be done by the freeze-pump-thaw method.

Below is the procedure for obtaining a 1D NOESY on the Bruker 500 Avance III. This is a combination of running in automation (IconNMR) and manually.

Procedure

1. On the 500's host computer, log into IconNMR as usual and run a proton experiment. Your upcoming 1D NOE experiments will go in the same folder with incrementing experiment numbers so name your file accordingly.

2. Open the proton data file in Topspin. Write down the exact frequency (in ppm) of the resonances that you will want to irradiate during your 1D NOESY.

3. For the next steps don't use IconNMR. Alternatively, in Topspin set up the next experiment with these commands:

    a. **iexpno** -to create a new dataset
    b. **rpar**, then choose dc_1D_NOE from the user file in the upper right corner. Click Read. Change solvent on next pop-up, and toggle the "Execute 'getprosol'" radio button to on, then OK.
    c. **o1p** – enter your ppm of the resonance to be irradiated
    d. **p12** – 10msec - 80msec – change as needed to get the irradiation specificity. 80msec will give the narrowest range, 10msec the broadest.
    e. **d8** - NOESY mixing delay - change if needed (0.3-0.5 for small molecules)
    f. adjust any other parameters as needed, like sw or ns
    g. **rga** – to automatically adjust receiver gain
    h. **zg** – to start the experiment

4. To process the spectra, type **ef**. Then phase the LARGE, selected peak down. The NOE peaks will be up (positive) for small molecules and down (negative) for large molecules. Artifacts are sometimes present and these will be anti-phase or dispersive (up and down character) but these will occur only for spins J coupled to the selected peak.

5. Repeat steps 3-4 if either p12 or d8 needs to be adjusted. Repeat as needed for any other resonances that you noted back in step 2. Subsequent **rpar**s will not be needed.