V. NOESY-1D

[updated: 26 June 2008]

1. Acquire a normal ^1H 1d spectrum, as described in Section I.

2. Make certain you turn the sample spinning to zero, or off. Having sample spinning during a NOESY1D will ruin the quality of the data. Check the shims for X and Y; if they provide some improvement, you should also 1st order shim XZ, YZ, XY, and X2-Y2.

3. Calibrate **pw90**, as described in Section II. This should be done for ***every*** sample prior to setting up and acquiring a NOESY1D.

4. If you do not have good information about proton T_1 values in this sample—if you have not previously performed a T_1 estimate on this, or a similar, sample—the obtain estimates of the slowest and longest T_1 values in this sample, as described in Section III.

5. Acquire a normal ^1H 1d spectrum, but with:
   - **pw=pw90** and **gain** set properly
   - **d1 = 3 ×** [longest T_1 for protons of interest]

     In other words, do not set **d1=3*T_1** where T_1 is the value for an aromatic proton that clearly will not be involved in the NOE you will be investigating.
   - **at=2 lb=0.5** (or **at=1 lb=1**, which are often good enough) provide sufficient resolution for NOESY1D measurements, since J-coupling is typically not of interest in these experiments.

6. Copy the fid to a new experiment: for example, if above was done in exp1, then:

   mf(1,2) jexp2 dsx↵

   would copy the fid in exp1 to exp2, join exp2, and ft and dscale.

7. Choose the multiplet of interest by surrounding it with a cursors box (left drag for cursor on left/high-frequency side, right-drag for 2nd cursor on right/low-frequency side):
   - try to keep the box symmetric about the multiplet
   - make the box "close-in" on the multiplet, but not "within" the multiplet
   - the selective pulse will grab portions of spectrum about 3× the width of the box, i.e., about 1× width on either side of the box
   - the UNITY can only do 10-12Hz selectivity; the INOVAs can go quite a bit smaller (≥ 4 Hz is fine), so for close-in multiplets, use an INOVA

8. Enter **NOESY1D↵** and make sure the box is still properly set
   - click on SELECT and then PROCEED
   - you can make multiple SELECTions prior to a proceed; this will array different mutiplets; it is easier to run other NOESY1D experiments in new exps to get other multiplets.
   - you should see the proper pw90 and power display one the top line after hitting PROCEED
   - if you make a mistake, never run the NOESY1D macro again on top of itself; return to step 5 above, and repeat on a new ^{1}H 1d spectrum.

9. Make certain **d1 = 3 × [longest T1 value for protons of interest]**

10. Make certain **nt = 64** or a multiple of eight.

11. Set **mix ~ [shortest T1 value for protons of interest]**
    - longer mix times can reduce J-coupled artifacts
    - make certain the spinner is off before acquiring data

12. The selected multiplet should show up negative; slight phase adjustments are often needed.
    - Positive peaks that show clean absorptive behavior are *positive NOE's*; these are common for compounds having MW < 1000.
    - Negative peaks that show clean adsorptive behavior are *negative NOE's*—common for compounds having MW > 3000—or protons undergoing chemical exchange with the selected proton.

      NOESY is given the name EXSY when chemical exchange is being studied; the experiment stays the same.
    - In a certain MW region (1000 to 3000 Da)—and for other protons depending on structure, solvent and temperature—protons can crossover between having positive and negative NOEs. ROESY1D should be considered for these protons/compounds, as ROE's are always positive, independent of MW or other sample conditions.