VI. TOCSY-1D

[updated: 30 June 2004]

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 TOCSY1D 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 TOCSY1D.
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. This is less important than for NOESY1D, but you still should have good idea of proton T_1 values to avoid repetition-delay artifacts.
5. Acquire a normal ^1H 1d spectrum, but with:
   - **pw=pw90** and **gain** set properly
   - **d1 = 1.5 ×** [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 TOCSY you will be investigating.
   - **at=4 lb=0** is common for TOCSY, as high-resolution is usually of interest.
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 INOVA can go quite a bit smaller, so for close multiplet problems use an INOVA
8. Enter **TOCSY1D↵** and make sure the box is still properly set
   - click on SELECT and then PROCEED
   - do not make multiple SELECTions with TOCSY1D, as mix will be arrayed.

- you should see the proper pw90 and power display one the top line after hitting PROCEED
- if you make a mistake, never run the TOCSY1D macro again on top of itself; return to step 5 above, and repeat on a new ^{1}H 1d spectrum.

9. **nt = 8** or **16** is usually sufficient. **nt=2** can be used, but 8 is better.
10. Use the default mix array if time allows.
    - **mix = 0,0.015,0.03,0.055,0.08** is a reasonable minimum array
    - TOCSY peaks will gain and lose dispersive character; the larger array is often useful in provide some spectrum that gives (as good as possible) adsorptive character to the multiplet you are interested in.
    - make certain the spinner is off before acquiring data
11. TOCSY works best with small or linear spin subsystems. It often is not very useful for small, highly saturated cyclic organic systems. COSY and its variants often work better for these kinds of compounds.