V. 1d NOE-Difference Spectroscopy

(7-July-04)

A. Discussion

- See the Bruker User's Guide introduction for discussion on this non-trivial, but powerful experiment.
- Sanders & Hunter also contain excellent discussions on NOE experiments (highly recommended)
- Use NOESY1D rather than NOEdiff; NOESY1D is a very significant improvement.

B. Critical Parameters

d1	–	relaxation delay; needs to be ≥ 3 T_1 (see later section for measuring T_1)
dpwr	–	must **not** be set above 14; typically 1-4 works well for saturating multiplets
dm='yyn'	–	typical, but **dm='nyn'** and **d2** can be used to vary saturation within fixed **d1+d2** delays
dof	–	set by command **sd** (preceded by placing cursor on desired multiplet)

C. NOE-Diff Acquisition

- read in a normal ^1H setup: MAINMENU SETUP NUCLEUS/SOLVENT
- SETUP SEQUENCE NOEDIFF will setup the following parameters:

  dn='H1' homo='y' dmm='c' dpwr=1 dm='yyn'

- place the cursor in the middle of the multiplet to be saturated and enter **sd**
- obtain enough data to determine
  i) if multiplet is being sufficiently saturated
  ii) if any other close-by multiplets are being effected by the saturation pulse; if any are, **dpwr** must be decreased until the close-by multiplets are all completely uneffected; this condition is essential–interpretation of the data becomes much more difficult if the chosen multiplet cannot be saturated independent of the other protons (if this happens, consider performing a NOESY or ROESY experiment instead of the NOE-diff)
  iii) currently, I do not know how to plot or save difference spectra if **dof** is arrayed (although this would seem to be the best way to acquire the data)
  iii) see cgfry about implementation of GOESY, which should be a superior experiment to NOEdiff; GOESY info should be available very soon

D. Calibration

- set **dpwr** low, and raise by 3 dB to no more than dpwr=14; use the lowest power that saturates the selected multiplet

E. Data Workup and Plotting

- bring in reference spectrum (with decoupler placed on baseline); make sure expansion and vertical scale are what you want
- make sure exp5 is saved and not in current use, then
- use **clradd spadd** to clear exp5 and transfer the current spectrum to it
- bring in next spectrum (in another exp if desired, but must have same expansion and vs; more precisely, **sp wp vs** must all be identical between spectra)
- use **addi** to compare and subtract spectra; see vnmr manuals for more info

VI. Homonuclear 1d Decoupling

(1-Jul-98)

A. Discussion

- HOMODEC has for the most part been replaced by COSY and cosy-variants, but still has utility for multiplets that are well resolved. HOMODEC can provide actual *J* couplings, whereas fast-COSY usually does not.

B. Critical Parameters

d1	–	relaxation delay; needs to be ≥ 3 T_1 (see later section for measuring T_1)
dpwr	–	must **not** be set above 24; typically 1-14 works well for saturating multiplets
dm='yyy'	–	correct for homodecoupling
dof	–	set by command **sd** (preceded by placing cursor on desired multiplet); can array this variable to obtain all homodec's in same experiment

C. Acquisition

- setup using SETUP SEQUENCES HOMODEC (or just type in **homodec** to run the macro)

  dn='H1' homo='y' dmm='c' dpwr=1 dm='yyy'

- array the **dof** values to acquire all in one experiment
- once decoupler is on, simply use **go** or **ga**

D. Calibration

- adjust **dpwr** (≤14) to reduce multiplet without spilling into close-by multiplets

E. Data Workup and Plotting

- workup as a standard ^1H
- **dssa** should be useful for plotting; see vnmr documentation