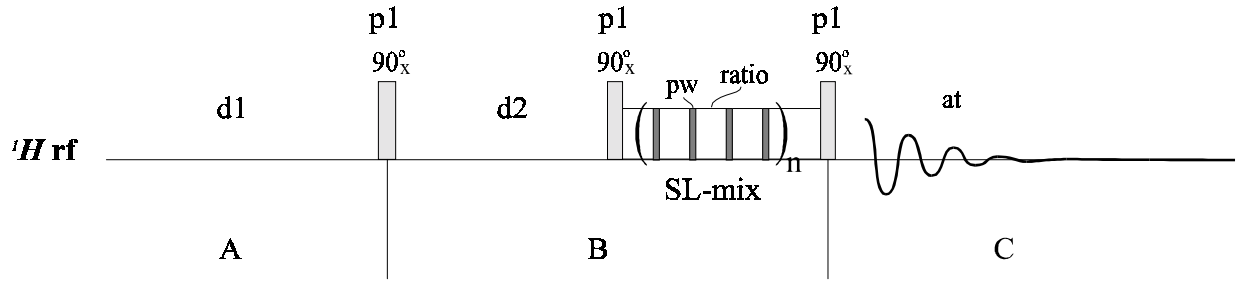
XII. ROESY – 2d NOE Spec.; Mixing via a Spinlock in the Rotating Frame

(17-Jul-00)

A. Discussion

- ROESY spectra will produce a maximum +0.5 enhancement for ^1H independent of MW. The experiment is still transient (as in NOESY), however, so enhancements are always smaller than 0.5. As always with NOE/ROE experiments, absence of a crosspeak does *not* confirm lack of proximity (see Sanders & Hunter for a good introduction on this subject).
- An ROE is produced by inverted magnetization created via chemical shift during t_1 (d2) and locked to the *y'* axis by the pulsatile spin-lock during the mix time. **pw** should be set at 30°, and **ratio** set to ~6 for **tpwr** ~ 52 on bbswg probe.

2d ROESY Spectroscopy (ulroesy)



B. Critical Parameters

p1, p1lvl = 90° pulse width at power **p1lvl**; recalibrate this parameter for roesy experiments
pw, tpwr = 30° pulse width at power **tpwr**; recalibrate this parameter for roesy experiments
ratio = number of **pw** with no rf between actual **pw** pulses; usually set ~6 (should be ≥5)
The spinlock field strength used in this type of experiment (Kessler spinlock) is:

$$B_{1sl} = \frac{1}{pw_{360}(\text{ratio}+1)}$$

Type **slroesy** to get the spinlock power for a particular **ratio** and **pw**.

ni = number experiments, or number of points in t_1 ; should be set ok by macro, time allowing; want F1 digital resolution **sw1/(2ni)** = 6 Hz/pt
nt = multiple of 2, multiple of 8 preferred
sspul = 'y' gives homopoil90-homospoil preceding **d1**
d1 = relaxation delay; set 2-3*T_1 (do not set too short, or will get very bad t_1 noise)
np = number of points in t_2, usually want ≥ 2048 since costs nothing but disk space and gives better resolution in F2
mix = mixing time; often is varied to provide build-up curves. Set close to T_1 should provide maximized crosspeaks; for high MW (>2000) NOESY is preferred.

C. ROESY Acquisition

- see NOESY section; adjust baseline similarly, and make certain to use actual ROESY sequence (do this by setting **ni=2**) when performing the **calfa** correction

D. Calibration

- calibrate **pw, tpwr, p1, p1lvl** for all ROESY experiments
- watch out for coupling partners that are centered in the spectrum; these will give TOCSY crosspeaks in a ROESY; in this case, change **tof** to get the coupling pair off-center

E. Data Workup and Plotting

- same as dqcosy; see DQCOSY section for phase-sensitive work-ups