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.

\[ B_{1sl} = \frac{1}{pw360 \times (\text{ratio} + 1)} \]

Type \texttt{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 homospoil90-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 \(\geq 2048\) 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.

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 \(\geq 5\))

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 \texttt{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