

Tips for quantitative integrations:  
(summarized by YB, June 2003, updated March 2008)

Acquisition hints for quantitative integrations:

- Measure the longest  $T_1$  and use  $5 \cdot T_1$  for the recycle delay
- Use a large sw to get a flat baseline
- Center your data in the middle of the spectrum (o1)
- Acquire a large number of dots (TD)
- Make sure the signal-to-noise ratio is high (NS)
- Use an optimal AQ time (fid going to zero at  $\sim 1/3$  of the screen)
- For decoupled measurements of X nuclei – AVOID NOE effects
- Repeat for a few different independent measurements

A recent reference giving more details, and recommending  $^{13}\text{C}$  decoupling:  
*J. Nat. Prod.*, **70** (4), 589 -595, 2007

Here is their list of "Factors" for quantitative NMR (qNMR) as opposed to the "normal survey proton NMR."

- (1) Don't spin, do shim (really well)
- (2) Remove the  $^{13}\text{C}$  satellites by decoupling
- (3) Relaxation delays so that, in Bruker-speak,  $aq + d1 \geq 5 \cdot T_1$
- (4) Spectral windows with about 2 ppm extra on each side of the data
- (5) Transmitter positioning to center the data
- (6) Pulse width selection to calculate the optimal pulse width according to the Ernst angle:  $\cos(\text{PW}_{\text{opt}}) = \exp(-aq/T_1)$ , where  $aq < T_1$   
Note: usually  $d1 = 0$ , but time is needed to minimize heating
- (7) Acquisition time with enough points to *i*) meet the Nyquist frequency condition, and *ii*) get a digital resolution of  $\sim < 0.2$  Hz/pt
- (8) Select sufficient repetitions, number of shots, for good si:no ratios
- (9) Receiver gain settings should be optimized: automatic is fine
- (10) Steady state: use dummy scans (ds) to establish an equilibrium
- (11) Set the  $^{13}\text{C}$  decoupling bandwidth and position with care

Processing hints for quantitative integrations:

- Do not use a window function, just do a straight fourier transform
- Be sure that the baseline is flat on both sides of the
- Include the  $^{13}\text{C}$  satellites in the integration
- Adjust the slope and bias of the integration
- Use the same limits of integration for all spectra  
(*i.e.* wmisc  $\rightarrow$  intrng, rmisc  $\rightarrow$  intrng)
- Use the same scale for all the integrations ("lastscal")
- Use deconvolution software, if lines are not resolved