Quick Instructions for No-D NMR
or
How to Take a Spectrum of Your Reaction Mixture

1) To lock or not to lock?

   A. The simplest way to do this experiment involves adding a capillary with an appropriate deuterated solvent (such as C6D6 or D6-acetone) to your sample so you can lock and shim normally. Melting point capillaries are useful for this or you can seal a Pasteur pipet. The good part is that once you seal them you can use them over and over again.

   B. If you elect not to add a capillary with a deuterated solvent to your sample, you must either do a gradient shim on the protonated solvent (see Gradient Shimming on the UI500NB handout) or shim on the FID. Either way, you should check with NMR Lab staff if you need any help. Gradient shimming is fast (~30 sec), FID shimming is not. Gradient shimming can be done on the ui500nb (hcn probe), ui600 (AutoX probe and hcn probe) and vxr 500 (QUADG probe). The procedures may not be exactly the same on each instrument.

   NOTE: If you have no lock solvent you MUST turn the lock off. Otherwise the system will ramp the lock frequency “looking” for a signal to lock onto, causing multiple lines in your spectrum.

2) Once you have locked (or not), and shimmed (or not), you should set up for the acquisition.

   If you did not lock, you should set up on the nucleus of interest (e.g., 1H or 31P) in CDCl3 because the Z0 for the lock will be set to CDCl3. If you don’t do this your chemical shift scale will be shifted by the difference in chemical shift between CDCl3 and the solvent you set up on.

3) Assuming 1H is your nucleus, set the following parameters:

   pw=7.5
   gain=0
   tpwr=46
   nt=1
   ga

Make sure you don’t see any error messages pop up on the top message line such as “ADC Overflow.” That indicates the signal intensity is still too large. You should NOT see the ADC Overflow message with these parameters.

References to No-D NMR: Hoye and Eklov:

*Org Lett* 6, 953
*Org Lett* 7, 2205
*Org Lett* 6, 2567