VII. Deuterium NMR on Varian Spectrometers

[updated: 25 July 2010]

1. Working in fully protonated solvent usually works best. The primary problem will be shimming without a lock signal. Use of a deuterated solvent (10:90% ^2H:^1H) can be used if long (>1hr) acquisition times are needed; this will enable magnetic field drift compensation using the ^2H lock. A ^1H spectrum will typically be acquired to provide referencing.
2. Hook up and view the LOCK-channel with the HP sweeper. Insert the proper cap rod for highest sensitivity ^2H detection [the lock channel can be used to simplify ^2H experiments—ask NMR staff for more info—if samples are at high concentration].
3. a) For bbext on the UNITY-500: detune the lock channel using the recessed screw—use ***only*** the non-magnetic screwdrivers in the lab!—close to the X-match rod (not at the outer rim, but toward the center of the probe)

   b) For hcx and bbswgo on the INOVA-500, or other older probes: turn only the outer black knob/capacitor at the bottom of the probe.
4. Hook up and tune the X-channel on the HP scope to ^2H; the resonance should not show asymmetry (or dual resonance) if the lock channel is sufficiently detuned.
5. For most samples containing protons, ^1H decoupling will be beneficial (for both NOE and decoupling). A ^1H spectrum will provide the best method of referencing the ^2H spectrum.

   – Tune the ^1H channel on the probe.
6. a) In the ACQI panel, turn LOCK off and

   b) set the LOCK POWER = 0

   c) Detach the lock cable from the probe.

   These steps should be performed, unless you need a lock on a ^2H solvent, for long experiments. If the lock is used, you will observe the lock transmitter signal, which can easily be mistaken for a real ^2H signal.
7. INOVA-500 only: remove the ^2H/lock-reject filter that is attached at the transmitter side of the X-rf cables from the probe: moving back from the probe along the X-cables is first the X-specific-filter, then another cable to two filters—one of which is the ^2H reject filter—attached to the preamp box next to the magnet leg.
8. Shim using one of the following methods:

Shim on the solute:

a) Acquire a normal ^1H 1d and expand around a solute singlet peak (or *known* simple multiplet), using nt=1 ss=0 d1~T_1, pw=pw90/3, gain optimized. Do not shim on a solvent peak; these are broadened by relaxation dampening too much to be used for shimming. For per-deuterated samples, a ^2H singlet can be used for shimming.

b) Expand so the singlet fills ≥30% of the display window, and type **gf**.

c) In acqi window, click on FID and shim on the spectrum.

Gradient Shimming is possible, and can be quicker once the interface is learned:

1st use requires a short setup, best checked by the NMR staff
[Unity: gzlvl=3200 d3=0,0.005 sw=80000 sqsinebell gzsize=5]
[Inova: read ^{1}H shimmap in for hcx probe]

a) connect proton cable for ^1H detection

b) start PFG shimming software: **gmapsys↵**

c) Unity-500 only: menu button: **SETUP**
menu button: **FIND GZLVL/GZWIN**

d) make a gradient map: menu button: **SHIMMAPS**
Unity only: menu button: **MAKE SHIMMAP**
Inova spec: menu button: **AUTOMAKE SHIMMAP**

e) let the shimmap finish (takes about 1min)

f) shim z-shims menu button: **AUTOSHIM**

g) always click on menu button: **QUIT**

h) may have to go into ACQI window and restart sample spinning

9. Acquire a ^1H spectrum. If ADC or receiver clipping occurs at **gain**=0 , try **pw**=1. If clipping still occurs, set **tpwr**=45. You need only enough to reference the spectrum. Save it with **svf** , and archive it with the ^2H spectra.

10. Move to a new experiment: e.g., if the ^1H spectrum is in exp1, do **jexp2↵** .

11. Read in ^2H,SOLVENT parameters, and acquire the ^2H spectra. For quantitative spectra, set **dm='nny' d1=2** (likely long enough). For best sensitivity, set **dm='yyy'** .

12. Reference the ^2H spectra using **xref↵** . Save as normal with **svf** .

13. When finished, you ***must***:

a) retune the lock channel on the probe

b) INOVA-500 only: re-insert the lock-reject filter on the X-channel

c) UNITY-500 only: reconnect the lock cabling with ^2H filter to the probe's lock port

d) insert and lock the $CDCl_3$ standard sample.