NUCLEAR MAGNETIC RESONANCE RESEARCH RESOURCE

Acquiring ^{2}H NMR Spectra

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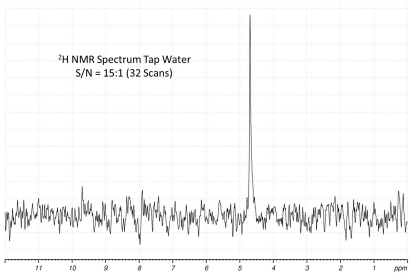
Before Beginning, Please Take Note of the Following…

- **Solvent:** Use non-deuterated solvents to avoid a ^{2}H spectrum where the solvent signal towers over (and possibly obscures) your solute signals.
- **Locking & Shimming:** ^{2}H NMR experiments must be acquired unlocked. In addition, a change in shimming procedure is required (instructions below).
- **Chemical Shift Referencing:** Since ^{2}H NMR must be acquired unlocked, chemical shift referencing cannot be performed in the traditional manner. Options for referencing your ^{2}H spectra include:
    - Use the Solvent ^{2}H Signal: Use the natural abundance ^{2}H signal or add a small amount of deuterated solvent to your protio solvent (~5%) for referencing purposes. The figure on the right shows the ^{2}H spectrum obtained from tap water in 32 scans. This signal is from the isotopomer $^1H^{16}O^2H$ (calculated concentration of 311 ppm or 16.3 mM).
    - Use the ^{1}H Signal of TMS from a previously recorded proton spectrum: an au program exists making this approach trivial.
    - Use a separate NMR sample containing the deuterated version of your protio solvent.



Instructions for ^{2}H NMR Experiments

(1) ***OPTIONAL*** If you have a sample containing deuterated solvent for chemical shift referencing purposes, insert this sample first and lock. Once the instrument is locked, eject the sample.

(2) Enter the command "**lockoff.ml**" to disable the lock channel. When complete (~ 10 s), an information window appears stating that the lock is off.

(3) Insert your NMR sample in the magnet.

******CAUTION******

Do not LOCK, SHIM or TUNE the PROBE while running ^{2}H NMR Experiments!

(4) Click NEW EXP in the button panel to create a new dataset for your ^{2}H NMR experiment. Select "**1d_2H**" from the experiment drop-down list.

(5) Shim by typing the command "**lctshim.ml**". This program will automatically perform a topshim using the most intense proton signal in your sample (usually from the solvent).

(6) Change "ns" if desired (64 is the default).

(7) Type "gpro" to ensure both the deuterium pulse width and power are up to date.

(8) Click START in the button panel to acquire the ^{2}H NMR data.

(9) ***OPTIONAL*** Reference your spectrum using the ^{1}H resonance frequency of TMS by typing the command "**xref.ml**".

(10) ***OPTIONAL*** If you have another sample for ^{2}H NMR, return to step 3 above.

(11) Eject your NMR sample from the magnet.

(12) ****IMPORTANT FINAL STEP**** enter the command "lockon.ml" before finishing. This returns the lock channel back to the default configuration for the next user.