IV. Direct X-nucleus Experiments

[updated: 25 July 2010]

A. Standard, NOE-optimized, decoupled experiments for positive γ, spin-1/2 nuclei such as ^{13}C, ^{19}F, ^{31}P, … but NOT for ^{29}Si, ^{15}N, ^{2}H, ^{109}Ag, etc.

[Nuclei having negative γ—^{29}Si, ^{15}N and ^{109}Ag being the most common examples—should not be run as described in this section. Use either INEPT or INVGATE (section IV.B or IV.D, respectively). INEPT (or DEPT) is by far the preferable method, having greatly enhanced sensitivity compared to INVGATE.]

1. Acquire a normal ^{1}H 1d spectrum, as described in Section I. The purpose here is to check that shims and therefore line shape are OK. Poor shims will reduce sensitivity, important to avoid with most X-detect experiments.
2. Tune the probe in the following way:
   - ***always*** tune the X-channel first; large changes in the tuning of this channel will change the tuning on the ^{1}H channel. If the nucleus is not one available from the menu on the HP oscilloscope, enter **sfrq** using FREQ, and increase the SPAN to ca. 80 MHz to assist in finding the reflectance dip on the scope. Reduce the SPAN to 4 MHz for final tuning.
   - follow tuning the X-channel with proper tuning on the ^{1}H channel.

   Tune the probe for ***every*** sample. Skipping this step can cause the ^{1}H channel to be mistuned. The high-power decoupler would then reflect potentially significant amounts of power back to the amplifiers, which could cause instrument damage.
3. Make certain cables are properly connected:
   a) UNITY-500 only: connect the ^{1}H cable from the probe to the DECOUPLER port.
   b) UNITY-500 only: connect the *X* cable from the probe to the *X*-OBSERVE port.
   c) All instruments: install the correct *X* bandpass filter in the *X* cable between probe and preamp connections.
   d) All instruments: check that the correct ¼-λ cable is installed.
4. Switch to another experiment: suppose the ^{1}H spectrum is in exp1. Do a **jexp2**↵
   a) Now do: MAIN MENU → SETUP → NUC,SOLV → [nucleus,solvent]. This reads in proper parameters for an *X*-nucleus experiment, but does not turn on the decoupler.
   b) Check that **dpwr=42** and **dmf=10000** [U500 as of July 2010; or whatever the proper values are, as these parameters can change].
   c) Use MAIN MENU → UWMACROS → DEC ON to turn on the decoupler. On the U500, the decoupler comes on as soon as an **su** (which should be done first), **go**, or **ga** is performed. On the INOVA instruments, the decoupler is only on during acquisitions. This means that on the U500, the decoupler ***must*** be turned off after the acquisition finishes (done automatically on I500).

5. **go↵** to acquire data. You can **wft↵** after each **bs** scans. **svf↵** can be done prior to (preferred) or following an **aa↵** to stop the acquisition.
6. Reference the spectrum to the ^{1}H spectrum using: **xref↵** . The ^{1}H spectrum has to be loaded and referenced in another experiment to use **xref**.

B. Quantitative spin-1/2 X-nucleus experiments (all nuclei: ^{13}C, ^{31}P, ^{29}Si, ^{15}N…)

1. Setup as described above in section IV.A.
2. Prior to starting the *X*-nucleus acquisition, set **dm='nny'** which removes the NOE enhancement, but retains ^{1}H decoupling.
3. Set **d1=5** for the first experiment. Acquire enough transients such that signal-to-noise is sufficient for your requirements; e.g., for kinetics data, likely want ±10% or better using integrals (which are significantly better than peak height).
   - Now acquire an identical experiment, but using **d1=15**. If the ratio of integrals is unchanged between the two experiments, then using **d1=5** is OK. (You can also try a shorter value, e.g., **d1=2**.) If the ratio changed, you need to increase **d1** again (e.g., d1=40). Continue until the ratio does not change, and then use the smaller **d1** value for your quantitative experiments.

C. Coupled spin-1/2 X-nucleus experiments (only positive-γ nuclei: ^{13}C, ^{31}P… but not ^{29}Si, ^{15}N…) [coupled DEPT or INEPT may be preferable]

1. Setup as described above in section IV.A.
2. Prior to starting the *X*-nucleus acquisition, set **dm='ynn'** which provides NOE enhancement (by $1+\gamma_H/2\gamma_X = 3$ for ^{13}C, 6 for ^{15}N).
   - Coupled spectra can be arduous in time to obtain, as long-range couplings will broaden the individual peaks within a multiplet, reducing signal-to-noise. See the facility staff for other, potentially better ways to obtain coupling constants.
   - DEPT or INEPT spectra can also be setup to obtain coupled spectra (by changing **dm='nnn'**), and provide somewhat better sensitivity (by γ_H/γ_X).

D. DEPT or INEPT experiments (all nuclei: ^{13}C, ^{31}P, ^{29}Si, ^{15}N…)

1. Setup as described above in section IV.A. MAIN MENU → SETUP → **NUC,SOLV** **must** be setup prior to the next step.
2. MENU → SETUP → SEQUENCE → **dept** or **ineptrd** ;or just type in the name.
3. See the Dept and Inept sections of VUG for more details about these experiments.