II. 1H Acquisition—Example Session

[cgfry: updated 8July2004]

- See "XwinNMR Acquisition Manual," Sections 1.4, 1.5 (list of acquisition parameter descriptions starting 1.5.2.4, pg. A-39), 1.6

a) Brief summary of commands

xwinnmr -r↵
edc or iexpno
setti
edte
rsh **cgf*
ii
lockdisp
lock
shim
rpar **cgf*
ii

eda
choosing new sequence maybe necessary → SAVE → eda
[PROSOL → TRUE] SAVE ; reads in probe-dependent parameters

(or gpro replacing above; make sure solvent is correct [case sensitive])

ased *check all parameters here*
eda *check "left out" parameters here, e.g., o1, td1, nd0, ...*
acqu
wob *UW macro nicer than Bruker's* wobb, just adds acqu in front
tune probe
calibrate 1H 90° pulse (at 360°) → adjust pulsewidths accordingly
rga
zg
efp

b) Detailed Setup

1. Use the Console window to start up xwinnmr: **xwinnmr -r↵**

2. Start your session using edc – edit the current data set name

   note: 14 character limit!! do not use spaces or special characters!

   I recommend you use a short descriptor followed by a date in the form 010511 for 11 May 2001. An example then would be: *sample1-010511*. Start in expno ***1*** procno ***1*** partition ***/u*** user ***fry*** where you replace *fry* by your own user login name.

You can alternatively use dir to bring up your datasets, and continue adding experiments to new expno's. You will likely then follow the dir with the use of edc to go the next available expno for the next experiment; the command iexpno does exactly this with less keystrokes.

I strongly recommend you ***not*** use a single dataset for multiple samples. I have done this, and it becomes quite confusing when you come back a few months later and try to figure out what expno matches which sample and experiment. In any event, use a notebook whenever working with XwinNMR.

3. Use ti parameter to comment the expno. You can edit the title, ti, using the command

   setti (or equivalently in our facility, use the UW macro: edti).

4. *1st time only:* Inside setres use initially *ZGsafe* set to off (or 'no'). Later on, you will need to change this setting to 'on' as otherwise multizg will not function without user interaction (defeating the purpose of multizg).
   - ☞ Set to **extended XwinNMR**.
   - ☞ Set your default editor (jot is a good choice).

5. To set the temperature, use:
   edte↵ brings up temp window; usually you will leave this open

6. Whenever starting a new session, use

   ```
   ii          ; initialize interface—maybe necessary to get lock to work
               ;      also sets up hardware properly
               ;      multiple ii entries in a row are needed after power outages
   ```

7. wsh and rsh work similarly to the AC/AM's, for writing and saving shims. However, once again Bruker forces all files from all users into a common directory, **/u/exp/stan/lists/bsms** (you can use **cdshims** in UNIX to move there), so you ***must*** append the suffix *.cgf* (use your own initials). That way:

   rsh **.cgf* will show just your shim files.

8. Enter lockdisp to open the lock display window.

   Lock using lock . Select the solvent, and the spectrometer should lock up.

   - ☞ Even if the spectrometer locks up automatically on your solvent, you must make certain you are on the correct lock solvent; otherwise the lock power will be set wrong, and shimming may be poor due to saturation.

9. To manually shim, work as usual similar to the AC's, with some additions:
   - Adjust the lock phase first to maximize the lock signal.

- With the spinner on (assuming a non-2D experiment), adjust Z1, Z2 and if necessary Z3. Push the ONAXIS button prior to clicking the Z buttons.
- Note that Z2 is not sensitive; you may want to turn off the FINE button when adjusting this shim. Bruker reduced the sensitivity of Z2 to stabilize the adjustment on our widebore magnet.
- If spinning sidebands are a problem, turn the spinner off and adjust all the low order X, Y shims. Click X with Z0 to get the X shims. Click X with Z1 to get XZ shims, etc.

10. You can gradient shim on the new 5mm probes by typing:
    gradshim↵ (we have not found gradshim to converge, so shim normally for now)
    - Gradient shimming on AVANCEs can currently only be done with protonated solvents, and only on the 5mm probes on our system. Skip to 8 if you are using a deuterated solvent, or the 10mm probe.
    - A gradient shimming window will open; the user should be *your_loginname*.
    - Click on the **Start Gradient shimming** button, and wait until a graph comes up and the SGI finishes pushing the new shim values into the shim unit. At this point, the shims should be pretty good. Do ***not*** adjust Z5 on this widebore 360 system.

11. Parameter sets can be read in rather than starting with a base sequence:

    rpar *cgf (lists just subset written by cgfry)
    rpar (lists all parameters sets saved by any user)

    wpar will save your parameters; all files from all users go into a common directory, **/u/exp/stan/nmr/par** (use **cdpar** in UNIX to move to this directory), so append the suffix initials so you can isolate just your files with the first rpar command above.

    ☞ all Bruker parameter sets use UPPER CASE letters; the facility also uses upper case with .UW appended; ***never*** use upper case for your parameter sets, and ***always*** use your initials as the suffix; that way the Bruker and facility sets will be obvious, and you will have a simple method of finding your parameters

    The following are known, good parameter sets:

PROTON	standard proton parameters using zg30
C13CPD	standard carbon parameters using zgpg30
C13DEPT*	standard C13 dept parameters
SI29.UW	Si29 parameters (mainly SF and SW)
H2.UW	H2 parameters
L6.UW	L6 parameters
L7.UW	L7 parameters
HOMODEC.UW	

12. For X-nuclei, you will have to change the pulse sequence unless you have a parameter or dataset written to recall. See the next section for more details.

13. Use eda and go to roughly the middle of the list; find PROSOL and change it to **TRUE**; then click on SAVE at the bottom of the screen; this will load in the probe-dependent parameters (such as P1/PL1 90° calibration). The rf values read in should be close, but do not assume they are correct!! Check all rf powers, especially all decoupler values.

14. Old data sets can be read in by typing:
    dir and clicking on the desired set.

    In Unix, you can get to your dataset by typing: **cddata**
    The path is **/u/data/*fry*/nmr**

    Make new datasets, or new experiment numbers with:
    edc

15. Use an Xshell window to look at pulse sequences:
    cdpp equivalent to **cd /u/exp/stan/nmr/lists/pp**

    ☞ We will provide a .cshrc file that you can copy that will provide various command "shortcuts" such as **cdpp** .

    cp *pulsesequence pulsesequence.cgf*
    jot *pulsesequence.cgf*

    ☞ always copy the sequence to your initials if you want to make changes or add comments; you will not have privileges to change the standard sequences)

    - all Bruker original sequences are kept in **/u/exp/stan/nmr/lists/pp.orig**
    - you can also use editors within xwinnmr by typing: **edpul**
      the default editor that is used can be changed in **setres**

16. A list of known, good sequences are provided in the next section. For now, we see in the **/u/exp/stan/nmr/lists/pp** directory:

    zg30 standard acquisition; p1,pl1 are 90° pulse, and 0.33*p1 is used as a 30° flip pulse in the sequence

    ☞ NOTE: pl1 is a "power level" stated in terms of attenuation applied in dB. This is similar to usage on the ACs.

    pl1 = -6 is ***maximum*** power, typical for hard pulses
    pl1 = 120 is ***minimum*** power
    pl1 = 16-18 is typical powers used for 1H decoupling

zghd same sequence homodecoupling set correctly

A listing can be brought up of the sequence with edpul *pulseprogname* or in UNIX using the unix editors (start with **cdpp** and then **vi *pulseprogname*** or similar).
You can see all pulse sequences by going into the ased or eda window and clicking on the down arrow next to the pulsesequence. You can also change it directly by typing pulprog↵ and typing in the new name.

17. Check the acquisition parameters by using:

    ased

    ased brings up a panel with pulse acquisition parameters specific to the pulsesequence chosen. If you change the pulse program within ased, exit and re-enter ased before changing other parameters.

    Then check other parameters (for reasons unknown not included in ased) using:

    eda

    o1, td1, nd0, and various other parameters have to be checked in eda or with the command line; ie. you will not find them in ased. Typically use DQD.

    It is recommended that the pulse sequence be open and checked while going through the parameters for the first time.

18. Check the probe tuning with

    acqu then wobb ;wobb is observed in "fid/acqu" window

    The sweepwidth can be changed with the WOBB-SW button. Stop the wobb with STOP. If you are performing an X-nucleus experiment (direct or inverse), the channels can be switched at the preamp box next to the magnet.

19. Parameter optimization can be done in three ways:

    a) Use gs and then acqu to start the go setup routine. An icon will appear that provides a sliding bar that can be used to adjust o1 or rg, for example. You can exit or stop the gs by click on the **STOP** button.

    b) Manually change p1 for ns 1 acquisitions and look for nulls in FIDs of FTs.

    c) Use paropt (Bruker's semi-equivalent to vnmr's array command) as follows:

       - Take a single scan and phase and expand properly.
       - Click on the DP1 button and hit return through the three query screens.

- Now enter paropt and answer the queries properly: e.g., p1 3 3 30 for a pulsewidth optimization using the sequence zg. Note that for zg30 you would need to use p1 9 9 30 to get equivalent results, since zg30 multiplies p1 by 0.33.
- paropt places results into procno 999. To return to your original experiment (assume it's in expno 1), enter re 1 1

20. Check all parameters, especially following the comments at the bottom of the pulseprogram listing. Note that Bruker may not be consistent in their use of pulse, delay or constant definitions. Do NOT assume similarly named sequences are written the same!!

    expt gives experiment time; typically aq d1 ns and td1 (for 2d exps) dominate times

21. Note the uncertainty in the description 2 paragraphs below, which would normally be recommended.

    For now, use rga to set the receiver gain [cgfry 8July04: rga is the best way to set the receiver gain!]. Note that on other Bruker and Varian spectrometers, rga can lead to very wrong settings of the receiver gain, so use with care!

    Use **zg** and **acqu** to start acquisition and switch to real-time fid observation to check the receiver gain level. Clipping occurs at the top and bottom of the unaltered acqu window (±12,000)??? [unsure about this!!] For a 2D sequence, watch both the first and second rows, don't rely on just the first row as a check. For COSY-type sequences, change d0 ~ 10ms so the magnetization is echoing back for an rg check (remember to set d0 back after adjusting rg).

22. Use zg and acqu (to observe 1st fid) to start acquisition. Transformation will not occur automatically; note the *finished* message in the bottom info bar.

    efp to transform and phase.

    tr to push data to disk to allow transformation during acquisition

23. Use edp to process the data in xwinnmr. For 2d data, the comments in the pulse program will tell whether the data was acquired TPPI, States-TPPI, or States.

    xfb will transform in both directions keeping all quadrants of the data.
    xfb n will transform, but not keep the imaginary components; much faster, but you won't be able to phase without retransforming here.

    Use the +/- buttons to turn on negative and both signs of 2d sets.

1d: use PHASE CUR and click middle button to define the toggle point, then click-hold the left button on PH0 and phase at the toggle point. Click-hold the left button on PH1 to 1st-order phase far away from the toggle point. pk will apply the previous phase correction.

2d: use PHASE CUR ROW and click middle button in 2d set; click on 1 to place in the 1 window; repeat for 2 and 3 windows. Then do PH0 and PH1.
Repeat complete process for CUR COL if F1 needs phasing.