Bruker AVANCE-360 User's Guide
for the UWChemMRF

by

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last revised: 2004.08.01

UWChemMRF User Guide for XwinNMR

I. Introduction

a) Account information

- passwd rules
  - do ***not*** use your email or 9th floor password; should be unique to CIC
  - use seven to eight characters, at least one non-alphanumeric
  - do not use any characters other than letters (case-sensitive), numbers, period, hyphen, underline
  - avoid simple substitutions: e.g., *b00tleg* is not a good password
- checkout requirements
  - all homework must be completed with 100% score before taking checkout
  - account locked-out one month after training if checkout not completed
  - account sharing will cause loss of access

b) Help

- See "The Interaction Between XwinNMR and Unix," pg. 4, 63 and 64
- All the Bruker manuals are accessible at the spectrometer under the XwinNMR Help menu.
- All the Bruker manuals are accessible on-line from our home page:

  go to: http://cic.chem.wisc.edu/nmr/main.html
  find: User Guides
  click on: Bruker AVANCE User Guides
- These documents are available in the same area of the web site.

Use the on-line and printed help manuals often and regularly. Asking questions prior to your having looked first yourself will not be well-tolerated.

c) Natoth SGI Unix – brief intro

- See "The Interaction Between XwinNMR and Unix," Part I; especially pgs. 5-7, 15-18, and 27-30 (note: much of the rest requires root privileges)
- .cshrc and aliases

dir	; **ls –laF** (at UNIX prompt only; different in Xwin)	
df –k	; check disk utilization	
cd	; standard csh—puts in home directory	
cddata	; only cd alias that is user specific	pg 79-80
cdshims	; shims directory	pg 70
cdpp	; pulse programs	pg 71
cdpar	; parameter files	pgs 69, 84
cdmac	; macros (simple command execution)	pg 70, 90
cdau	; automation files (c-like)	pg 69, 85-89
cdgp	; gradient programs	missing on pg 69

d) File structures and naming conventions

- See "The Interaction Between XwinNMR and Unix," Part II; pgs 66, 69, 70

Filenames	14 letter limitation; edc
Data locations and structure	pgs 79-80
Shim files	pg 70
Parameter files	pgs 69, 84
Pulse programs	pg 71
Macro files	pgs 70, 90
Automation files	pgs 69, 85-89

e) XwinNMR and setres

- See "The Interaction Between XwinNMR and Unix," Part II; pg 91

ZGSafety	; 'yes' initially, but *must* be set 'no' for **multizg**
Default Editor	; use "jot"
XwinNMR	; set to "Extended"
Apply	; will correct color/display problems

f) Editors

- See "The Interaction Between XwinNMR and Unix," pg. 72, 79

edc	; edit current (for new data acquistion)
eda	; edit acquisition parameters
ased	; automation setup editor (pp dependent)
setti (edti; UW macro; also edinfo)	; edit title (useful for data commenting)
edte	; edit temperature (temp panel)
edhead	; edit probehead (for probe changes)
edp	; edit processing parameters
edg	; edit plotting parameters

g) Modern High-Field Spectrometers

Linear Amplifiers ; see edacb

Bruker (stated in attenuation, not power!!)		Varian
-6dB	high power proton bb probes	63
0dB	high power X	58
≤30dB	presaturation	24
≤40dB	homodecoupling	18
≤16dB	highpower decoupling	41
≤10dB	highpower spinlock (check for update before using)	41

Tuning probes ; see Acquisition Manual 1.5.4, pg A-60

wobb	; needed for changes in temp for 1H ; required for all X-nucleus experiments
wob	; UW macro, does an acqu prior to wobb

h) Starting and Stopping Acquisitions

- See "XwinNMR Acquisition Manual," Section 1.7
- do **not** use quicknmr or run (they are not setup)

```
zg        ; zero data then go
go        ; do not zero data (ie., add to current)
tr        ; transfer data for processing
halt      ; preferred method for ending acquisition
stop      ; immediate termination of acquisition, not as safe as halt
suspend   ; suspend and resume only if pulse program supports (usually not)
resume    ; resume only if supported in pulse program
kill      ; will terminate active processes (e.g.,
```

i) Plotting Data

- See "XwinPLOT manual" on cic website
- PLOT button works fine in XwinNMR (but pretty limited)
- type xwinplot to start up very nice plot editor
  - main customization is *right-click* on object and select *1D-2D-edit*

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.

III. X-Nucleus Acquisitions—Example Session

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

a) General Theme for X-Acquisitions

- Setup normally (edc, edhead, edte, lock, shim) until need to read in parameters.
- Choose a parameter set matching observe nucleus, e.g. rpar P31CPD .
- Use eda, ased, or pulprog↵ to change to desired pulsesequence, then SAVE.
- Use eda → PROSOL → TRUE → SAVE (or equivalently gpro) to read in ***some*** but not necessarily all probe dependent parameters.
- Check parameters carefully with ased then eda comparing to listing of pulsesequence (using **cdpp → jot** or edpul).
- Check probe tuning with wob .
- Acquire data, using tr to check signal-to-noise.

b) Detailed Setup for X-Acquisitions

1. Use the Console window to start up xwinnmr: **xwinnmr -r↵**
   ; **-r** removes hung sessions

2. edc or iexpno – edit the current data set name

3. edhead – check that correct probe is defined

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

☞ use ii to initialize interface (if error messages appear, redo ii until they go away)

5. Enter lockdisp to open the lock display window. Lock using lock . Select the solvent, and the spectrometer should lock up.

6. Shim normally; best to check shims in a 1H spectrum. Poor shims will reduce sensitivity of any X-nucleas experiments, so good line shape is still highly desired.

7. 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; ***never*** use upper case for your parameter sets, and ***always*** use your initials as the suffix; that way the Bruker sets will be obvious, and you will have a simple method of finding your parameters

8. The following are known, good parameter sets:

PROTON	standard proton parameters using zg30
C13CPD	standard carbon parameters using zgpg30
C13DEPT*	standard ^{13}C dept parameters
P31CPD	standard ^{31}P parameters using zgpg30 (o1 not checked)
N15IG	standard ^{15}N parameters using zgig (o1 not checked)
N15INEPT	standard ^{15}N parameters using ineptrd (o1 not checked)
SI29IG.UW	UW ^{29}Si parameters starting with zgig
H2.UW	UW ^{2}H parameters
LI6.UW	UW ^{6}Li parameters
LI7.UW	UW ^{7}Li parameters
COSYGS.UW	UW 2d gradient COSY
COSY90SW	2d non-gradient COSY (for 10mm probe)
ghsqcse.UW	UW 2d sensitivity-enhanced 1H-13C 1-bond hsqc experiment
gChsqc.UW	UW 2d gradient 1H-13C 1-bond hsqc
gNhsqc.UW	UW 2d gradient 1H-15N hsqc (long-range probably next?)
ghmbc.UW	UW 2d 1H-13C long-range hmbc experiment

Listings of parameters sets in Unix can be accomplished with the **-d** qualifier:

cdpar
dir -d C13* or
ls -d S*

9. 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 (/u/data/cgfry/nmr**)
Make new datasets, or new experiment numbers with: edc

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

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)
- you do not need to copy sequences if you just want to see the listing
- all Bruker original sequences are kept in **/u/exp/stan/nmr/lists/pp.orig**
- you can also use editors within xwinnmr by typing: edpul *pulsesequence.cgf*

11. The most important sequences for X-nucleus acquisitions are:

zg30 ; not recommended, with no decoupling, no NOE, no PT

☞ zgpg30 ; standard decoupling sequence → with 30° pulse and NOE growth during d1

zgpg ; same as zgpg30 except with 90° pulse

zg0pg ; same as zgpg expect uses p0 @ pl1 for variable angle flip

zgdc30 ; same as zgpg30 except with a single power level for decoupling

zggd30 ; gated decoupling → with 30° pulse, coupled spectrum with NOE buildup during d1

☞ zgig30 ; inverse gated → with 30° pulse, for quantitative observation of decoupled X-nuclei, no NOE buildup; d1 ≥ $3\times T_1(X)$

☞ ineptrd ; inept+ → with decoupling:
cnst2 = J(XH)
cnst11 = $2\pi[\sin^{-1}(1/\sqrt{n})]^{-1}$ (rad)
= $360°[[\sin^{-1}(1/\sqrt{n})]^{-1}$ (deg)
where n=# ^1H involved in J(XH)
see http://cic.chem.wisc.edu/nmr/Guides/BUG/PT.pdf

☞ ineptpnd ; coupled inept+ → same as ineptrd with no decoupling during acquisition

ineptnd ; coupled inept → similar to ineptpnd but shorter; use if T_2's are short

☞ dept ; standard dept → p0 controls PT via last pulse on ^1H channel
cnst2 = J(XH)
p0 is set relative to p1 @ pl1 (^1H 90° pulse)
p0 = $\sin^{-1}(1/\sqrt{n})$ (rad or deg)
= p1/90° × [$\sin^{-1}(1/\sqrt{n})$ (deg)] (μs)
where n=# ^1H involved in J(XH)
see http://cic.chem.wisc.edu/nmr/Guides/BUG/PT.pdf

dept45 ; standard dept with final 45° pulse preset

dept90 ; standard dept with final 90° pulse preset

☞ dept135 ; standard dept with final 135° pulse preset

deptcp ; same as dept but with ^1H composite 180° pulse

☞ deptnd ; same as dept but no decoupling during acquisition, best for short T_2's

deptppnd ; dept++ with no decoupling during acquisition; less distortion than deptnd

☞ decp90 ; sequence to calibrate decoupler 90° pulsewidths
or use dept90 and minimize a CH_2 group

12. Chose the pulsesequence you need to use:

zgpg30	; for standard decoupled spectra with nuclei having positive γ (e.g., ^{13}C, ^{31}P, ^{19}F, ^{2}H) do not use for nuclei with negative γ (e.g., ^{29}Si, ^{15}N)
zgig30	; for quantitative spectra, or when no ^{1}H couplings or NOEs are available
zggd30	; simple coupled spectra; **deptnd** or **ineptpnd** should work better for coupled spectra
dept	; for ^{13}C reduces distortions arising from range of J_{XH}'s
ineptrd	; for ^{29}Si (seems to work better than dept?)

Make the change 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.

13. Use PROSOL → TRUE → SAVE or gpro to read in probe dependent parameters.

14. The facility will keep the most up-to-date pulsewidth calibrations in the probe-solvent files which can be viewed with solvloop. We will also keep a log of calibrations, which will include dates and temps for the calibration in a file:

probe-calibrations.txt

in the shims directory at: **/u/exp/stan/lists/bsms** (**cdshims** alias to move there).

15. Check the acquisition parameters by using: ased

Then check other parameters not included in ased using: eda

It is recommended that the pulse sequence be open and checked while going through the parameters for the first time (e.g., **cdpp jot *pulsesequence*** in UNIX window).

☞ use ii to initialize interface

16. Check the probe tuning with wob

Always tune the X-channel first, then the 1H channel afterward.

17. Perform any parameter optimization required.

18. 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 similar named sequences are written the same!!

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

19. For now, use rga 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!

20. 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

21. 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.

IV. 2D NMR on the AVANCE Spectrometer

[cgfry: updated 1 Aug 2004]

a) The Philosophy Used to Setup 2D Exps on the AVANCE-360

Bruker's XwinNMR unfortunately does not allow preset parameters, e.g., from a ^1H 1d experiment, to be easily passed to a new experiment. All multidimensional experiments are therefore setup here by a "brute-force" method as follows:

- A parameter set containing a valid set of parameters is read in using rpar .
- Standard probe rf parameters are read in by setting PROSOL to TRUE in eda .
- The sweepwidth, center frequency, and referencing parameters must be set by manually copying them from the appropriate 1d data set into the 2d data set. eda is the best place to perform these changes, as this panel shows the parameters for both dimensions.
- All individually calibrated parameters must be copied to the 2d data set, such as:
  - RG
  - the 90° ^1H pulsewidth (usually p1 @ pl1), and
  - the repetition delay (d1) based on the proton T_1.
- It is recommended that the user open the associated pulse sequence, and carefully check that *all* parameters are set as described in the comments section of the sequence (especially for long experiments). The sequence is best read/opened as follows:
  - Find the pulse sequence named by looking at the first line in either the eda or ased panel.
  - In a unix window, enter jot *filename*↵

b) COSY 2D Experiments

- Setup normally: edc, edhead, edte, lock, shim, wob.
- Acquire a standard 1H 1d dataset. Optimize the sweepwidth and O1 and reacquire.
- Use edc or iexpno to move to a new experiment #, and read in the cosy parameters:
  - on 5mmbbo, use rpar COSYGS.UW all↵
  - for the 10mmbbo, use rpar COSY90SW all↵ (or COSY45SW)
- Copy SW, O1 and RG from the 1H 1d dataset into the 2d.

  SW1 = SW , and
  O2 = O1 .

  Do ***not*** use RGA on a cosy dataset, as the 1st row is lower in intensity than later rows.

- Check that TD1 in eda is sufficiently large to provide the desired J-coupling observation. TD1 = 128 will be sufficient on the 360 in most cases.
- Set d1 ~ T_1 of the longest protons of interest.
- Set NS to 1, 2, 4, or multiples of 8; 1 or 2 should be sufficient unless the sample concentration is very low.
- Start the acquisition, and look at the first row acquisition in the acqi window; protons should be observed. Transform the 1st row if necessary; increase NS if necessary to provide sufficient sensitivity.
- expt will give an estimate of the total time of the experiment.
- For processing, go into edp: SI1 must equal SI for symmetrization.
  - Use xfb and sym to transform and symmetrize the data.
  - Make sure SR = SR1 equals the SR value in the 1H 1d to get the referencing correct in both direct and indirect dimensions.

c) HSQC 2D Experiments

- Setup normally: edc, edhead, edte, lock, shim, wob.
- Acquire a standard 1H 1d dataset. Optimize the SW and O1 and reacquire. Properly reference the spectrum.
- In another experiment, acquire a 1 scan ^{13}C (or ^{31}P, etc.). Optimize SW and O1 based on knowledge of the compound; limit to only 1-bond protonated carbons if only hsqc will be acquired. You might expand to include all quaternary carbons if a long-range hmbc will also be acquired.
- Use edc or iexpno to move to a new experiment #, and read in the hsqc parameters:
  - on 5mmbbo, use rpar ghsqcse.UW all↵
  - for the 10mmbbo, see Charlie for assistance in locating a non-gradient version of the experiment, and learning how to optimize it.
- Copy SW, O1 and SR from the 1H 1d dataset into the 2d for these exact same parameters. SR can be typed in, or found on the edp page.
- Copy SW, O1 and SR from the 13C 1d dataset into SW1, O2 and SR1. SR1 is found on the edp page.
- Check that TD1 in eda is sufficiently large to provide the desired resolution in 13C chemical shift. TD1 = 128 will be sufficient on the 360 in most cases, although where some carbons are close, TD1 up to 512 will be better.
- Set d1 ~ T_1 of the longest protons of interest.
- Set NS to 2, 4, or multiples of 8; 2 should be sufficient unless the sample concentration is low.

- Do ***not*** use RG from the 1H 1d dataset here. Only 1.1% of the protons, those attached to 13C, will be observed.

  Use RGA to properly set RG . [The probe must be properly tuned!]
- Transform the 1st row of data; increase NS if necessary to provide sufficient sensitivity to see some of the 1H directly.
- expt will give an estimate of the total time of the experiment.
- For processing, go into edp: set SI equal to 1/2×TD (no zerofill), and SI1 ≥ TD1 (at least one zerofill; two is common).
  - Use xfb to transform the data.
  - Make sure SR and SR1 are set properly form the 1H and 13C 1d datasets, respectively, to get the referencing correct in both direct and indirect dimensions.

d) HMBC 2D Experiments

- Follow the instructions for HSQC above, but with the following changes:
  - Make certain the 13C 1d SW is large enough to include all quaternary 13C.
  - Use rpar ghmbc.UW all for the 5mmbbo probe. HMBC on the 10mmbbo probe is not recommended; gradients very significantly improve the quality of this experiment.
  - Always calibrated the proton channel 90° pulse width for HMBC.
  - See the pulse sequence listing to insure that the long-range J-coupling parameter is set properly.
  - Always use NS equals 8, or a multiple of 8.