

NMR Quicknotes 2D

A guide to COSY, NOESY, J-resolved and HETCOR spectroscopy at Haverford College.

Rob Scarrow, printed 2/16/07 9:06 AM

Meaning of symbols in this document

CLB/CMB/CRB Click using left/middle/right mouse button.

sw - sfo1

CLB on button within xwinnmr.

stand by

Push button on SCM unit.

zg

Type at the bottom of xwinnmr window (followed by Enter).

EXPNO 5

Type "5<Enter>" in box labeled EXPNO in dialog box.

1) Temperature, Lock and Shim

Follow instructions for "Lock and Shim" on 1-D Quicknotes. (Optional – turn on temp. control)

2) High-resolution ¹H spectrum

It is helpful to plot a ¹H spectrum next to the proton shift axes in 2D NMR. Follow instructions on 1-D Quicknotes (use expno = 1).

After processing, choose a display region including all peaks but extending only 0.5 ppm beyond them.

sw - sfo1

Sets frequency offset (O1) and sweep width (SW) to cover the displayed region. Record the values of O1 and SW that appear.

rg

Record the value of the receiver gain. Hit return.

sf

Record SF (the calibrated frequency of the TMS peak).

3) Before HETCOR or HMQC: ¹³C DEPT135

For 2D spectral techniques involving ¹³C, the DEPT135 is recommended for plotting next to the carbon shift axis. Follow instructions on 1-D Quicknotes (use expno = 4).

After processing, choose a display region including all peaks but extending only 5 ppm beyond them.

sw - sfo1

Record O1 and SW for ¹³C.

sf

Record SF for ¹³C.

4) Set-up parameters for acquisition

edc

EXPNO 5

rpar

Read in parameter set.

Customized parameter sets "hetcor", "hmqc", "jres", "cosy90", "cosydqf", and "noesy" follow the upper-case Bruker-supplied parameter sets in the listing.

copy all

(OK to the message)

eda

Enter the following values in order, using values written down in steps 2 & 3. Start near the bottom of the eda scrolling window and work upwards.

SOLVENT ___ (CRB to select solvent)

PROSOL true (CLB to set to true)

O1(F2) ___ HETCOR: **o1** from ¹³C (step 3).
Otherwise, **o1** from ¹H (step 2)

O2(F2) ___ HMQC: **o1** from ¹³C. Otherwise,
o1 from ¹H

SW(F2) ___ HETCOR: **sw** from ¹³C.
Otherwise: **sw** from ¹H

SW(F1) ___ HMQC: **sw** from ¹³C.
J-res: 0.40 ppm.

Otherwise: **sw** from ¹H.

RG(F2) ___ HETCOR: leave at max value
Otherwise: **rg** from ¹H (step 2)

edp

SF(F2) ___ **sf** from ¹H (HETCOR: from ¹³C)

SF(F1) ___ HMQC: **sf** from ¹³C.
J-res: same as **SFO1(F1)**.
Otherwise: **sf** from ¹H.

edg

EDPROJ1 ed CLB on the "ed" box to edit the disk, user and exp. names, expno, and procno (=1) for the plot along the f1 axis
HMQC: expno = 4 (DEPT135)
otherwise: expno = 1 (¹H).

EDPROJ2 ed Do the same to specify the plot along the f2 axis
HETCOR: expno=4 (DEPT135)
otherwise: expno=1 (¹H).

5) Acquisition of the 2D spectrum.

Turn spin off (2D spectra are usually obtained without spinning).

lockdisp Check non-spin shims X&Y

expt Gives time required for data acquisition. Write this down.

If you wish to only collect one 2D dataset, then type

zg Acquire data for one data set.

If you wish to collect two or more 2D datasets, type

multizg Indicate how many 2-D spectra you will run in all.

Example: if you indicate you wish to collect 4 spectra, the computer will copy the acquisition and processing parameters from expno = 5 to new expno's 6, 7, & 8. It will immediately start collecting data for expno = 5.

While data is being collected, return to step 4, but use expno = 6, 7, 8 etc. for different 2D techniques.

Add up the "expt" for each expno and be sure the total stays within your time slot.

6) Data processing

Data processing is typically done later, when no one else is using the NMR (data processing can take place in the background while 2D data is being collected, if the same login user account is used).

xfb FT in both directions

For J-resolved only:

tilt 45° tilt of spectrum

For NOESY spectra, as well as many of the Bruker-supplied experiments, you must now phase the spectrum. This is described on page 2.

7) Viewing and Plotting 2-D spectra

*2 /2

CLB on these buttons to change which contour levels are shown on the screen.

colors

Hold left mouse button down and move mouse up and down to change the number of colors used to represent different intensities.

+ / -

Depending on what kind of spectrum processing has been done, you may have both positive and negative contours (peaks and valleys, shown in red and blue) to display. **CLB** on this button to change whether peaks, valleys, or both are displayed.

If you desire an expansion, **CLB** on spectrum and **CMB** successively on opposite corners of the region you wish to display. Then **CLB** to release the cursor.

CLB to zoom in on region just defined.

defplot

Usually default answers are fine.

[edo](#)

Check that **CURPLOT** is correct.

[view](#)

Preview the appearance of plot

[plot](#)

Plot should print out.

8) Conserving Disk Space

The FID's for 2-D spectrum are big and should be deleted within a week or so of acquisition (use "dela"). The processed spectra are even bigger, and should be deleted before you leave the computer:

[del2d](#)

Computer thinks several seconds before presenting you with a list of 2-D processed spectra. Select (**CLB**) yours. If it doesn't say "deleting data only" then **CLB** on [mode](#) until it does.

[execute](#)

9) Phasing (HMQC and NOESY)

phase

+ / -

CLB twice on this button to show pos. and neg. contours.

*2 /2

CLB on these buttons to change which contour levels are shown on the screen.

row

CLB on this button (in the second group of buttons on the left) and then **CMB** at the correct position on the 2-D spectrum to select row with leftmost peak.

+ -

These buttons, again in the second group of buttons on the left, fine-tune the row spectrum. The row spectrum should appear in white toward the bottom of the 2-D spectrum. Tweak the position of the row to get the largest peaks in the row spectrum.

mov 1

CLB on this button (in the third group of buttons) to move the row spectrum to green area #1 on the right of the screen

Repeat last three steps to select rows with peaks at middle and right of spectrum. Move them to areas 2 and 3 on the right of the screen.

cur 1

CLB on this button, then **CMB** on leftmost pk. in spectrum 1 (ref. pk.)

PH0 PH1

Drag up and down on [PH0](#) to phase the ref. pk. at left of spectra, and then on [PH1](#) to touch up peaks at right of spectrum

return

Choose "Save & Return" option

OK

Start xf2p (phase correction)

Now repeat the steps above, but select three **columns**. These should require only a minor phase correction (xf1p).

10) By-passing phase correction

If you can't get the knack of phasing, you may try a magnitude calculation, which is used by default anyway for the cosy and jres parameter sets.

[edc](#)

[PROCNO 11](#)

set up a new procno

[edp](#)

[WDW sine](#)

(in both F1 and F2)

[SSB 0](#)

(in both F1 and F2)

[PH\(F2\) no](#)

[PH\(F1\) mc](#)

[xfb](#)

11) Background t₁-noise subtraction (useful for COSY, HMQC etc.)

utilities

In the F1-axis block of buttons, **CLB** on

ext

Should show DEPT 135. Scale with [^2 v2](#) button)

Choose ¹³C ppm range for background subtraction. Should have no cross peaks. 70-100 ppm is often useful, and is assumed for the example below.

In the F2-axis block of buttons, **CLB** on the calc button that is next to the part button:

part [calc](#)

The cursor should move to the spectrum. **CMB** twice to define the upper and lower limits of the partial projection to be calculated. For each ppm along the x (f2) axis, the largest valued noise peak between these two limits will be calculated. A dialog box will appear after the second click.

[2](#)

Type new PROCNO in place of "type new name"

[return](#)

To exit the utilities routine.

[edc2](#)

[PROCNO2 2](#)

[sub2](#)

Subtracts the partial projection from your 2D-NMR. Removes most "t₁ noise".

12) Symmetrization

COSY, NOESY and JRES spectra should be symmetrical. There are symmetrization commands in the "Proc" menu. Use these with caution, and always report if a spectrum is symmetrized.