

HMQC/HSQC Quick Reference – Best Sensitivity on Artemis/Hades

(See Tips and Tricks at the end of this section before starting)

NOTE: HSQC picks up one bond H-C coupling (analogous to HMQC). Generally, it gives a much cleaner spectrum than the HMQC experiment...however, it can appear less sensitive than HMQC.

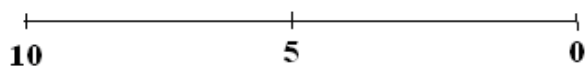
1. *If you already have a ^1H spectrum and know your **sw** and **o1p**, you can skip to step 2.* Setup and obtain a 1-D proton spectrum. Be sure to check the tuning and matching of the probe – see instructions in this binder. Determine the optimal spectral window around your peaks of interest allowing for ~ 0.5ppm on either side of your peaks. If your resonances fall between 1-8 ppm, you should select a **sw** of 8 (from 0.5ppm to 8.5ppm). The center of your spectrum is called **o1p** and for the above example it would be 4.5ppm. Record **sw** and **o1p**.
2. If you already have a ^{13}C spectrum for your sample, you should select an **sw** and **o1p** that will include all the protonated carbons allowing for ~10ppm at both extremes. Example: Your ^{13}C spectrum has peaks between 20ppm and 120ppm, you should select an **sw** of 120ppm (which will span from 10ppm to 130ppm by setting **o1p** at 70ppm). If you do not yet have a ^{13}C spectrum, you can either acquire one now or after you run the HSQC, but you will not be able to optimize the **sw** and **o1p** for the ^{13}C dimension without a spectrum (use the default values unless you can predict the limits from your structure).
3. Type **edc** [enter] and change the experiment number to 3. Type **rpar** [enter] and select **HSQC_EDITED** (this experiment is multiplicity edited such that CH & CH₃ signals will have an opposite phase from CH₂ signals-analogous to DEPT135 result), **HSQCGP** or **HMQCGP** [enter], **copy all** [enter] (Click **ok** or **seen** to any boxes that pop up after the **copy all** command). *NOTE: On Artemis, you will need to select the versions that have the suffix **_BROWN**...if you don't see it in the list, make sure you are in the directory that ends with **/user**.* Type **eda** [enter] and change **sw & o1p** (in F2 column) and **sw & o2p** (in F1 column) values to those determined optimum for **sw/o1p** in the ^1H and ^{13}C experiments, respectively. Turn **off** the sample spinning (either by pushing the button on the BSMS console – top left – or in the shim panel of the bsms display). Touch up **z** and **z2**. If you have not done so already, tune and match the probe following the directions in this manual (On **Zeus** and **Hades** – just type '**ATMA**', on **Artemis**, use the '**wobb**' command). When the tuning and matching is complete, type **rga** [enter]. Type **zg** [enter] to begin the experiment.
4. Type **xfb** [enter] to process the 2-D data any time during the acquisition. *NOTE: Only the HSQC experiments are phase sensitive. Consult the '2-D Phasing Guide for Topspin' if you wish to phase your spectrum.* Type **abs1** [enter], and **abs2** [enter] to perform a baseline correction on your spectrum. You can stop your acquisition before it finishes if you have already resolved your cross peaks of interest. Just type **halt** [enter] and **xfb** [enter] to process the latest scans. The spectrum will be saved. *NOTE: If the scale is off in either*

dimension of your 2-D plot, type **edp** and make sure the offset values are correct! If they are not, change the **offset** in F2 and F1 to the values recorded for **offset** in your optimized 1-D spectrum for the ^1H and ^{13}C experiments, respectively. NOTE: Due to a bug, you may have to go in and change the offset value multiple times.

- If you do not see any meaningful correlations at all, go back and double check your **o1p** values in the **eda** menu.

TIPS and TRICKS FOR 2-D EXPERIMENTS

example



SW = Spectral Width = 10ppm

o1p = 5 ppm (center of your spectrum

offset = 10ppm

- Select an **sw** and **o1p** that are easy to remember. If your ^1H spectrum has peaks from 2.5 to 7.5ppm, you would select an **sw** of 6 and an **o1p** to 5. The **offset** value in the processing parameters should always be the most downfield ppm value, in this case **8**.
- If you already have a proton and carbon spectrum for the sample on which you wish to perform a COSY, HMQC/HSQC, or HMBC, just determine the optimum **sw**, **o1p**, and **offset** values for those spectra and plug those values into the appropriate 2-D parameter set. You do not need to rerun the ^1H and ^{13}C spectra. Come see me if you want to learn how to incorporate the traces from old spectra into your 2-D dataset for processing.
- Linear prediction is a useful tool for improving the resolution in the indirect dimension of any 2-D experiment. To use this, click the ProcPar tab and scroll down to LPfr and increase the # of output points for LP from 0. Example: Your experiment is running and you have acquired 32/256 steps in the indirect dimension. Increase the # of points to 96 and you may be able to stop your experiment sooner. The # of output points should not exceed 3X the actual # of steps in your indirect dimension.

Fourier transform			
TDeff =	0	0	# of fid data points used by ft
STSR =	0	0	First output point of strip transform
STSI =	0	0	Total # of output points of strip transform
ME_mod =	no	LPfr	Linear prediction for ft, xfb, ...
NCOEF =	0	32	# of LP coefficients
LPBIN =	0	0	# of output points for LP
TDoff =	0	0	# of back-predicted points

An arrow points to the '32' value in the NCOEF row.