

# HMQC/HSQC - Quick Reference for ZEUS/ARTEMIS/HADES

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

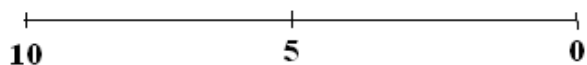
*NOTE: HSQC picks up one bond H-C coupling (analogous to HMQC). Generally, it gives a much cleaner spectrum than the HMQC experiment...however, it can appear less sensitive than HMQC.*

1. If you already have a  $^1\text{H}$  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  $\sim 0.5\text{ppm}$  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  $^{13}\text{C}$  spectrum for your sample, you should select an *sw* and *o1p* that will include all the protonated carbons allowing for  $\sim 10\text{ppm}$  at both extremes. Example: Your  $^{13}\text{C}$  spectrum has peaks between 20ppm and 180ppm, you should select an *sw* of 180ppm (which will span from 10ppm to 190ppm by setting *o1p* at 100ppm). If you do not yet have a  $^{13}\text{C}$  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  $^{13}\text{C}$  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 & CH<sub>3</sub> signals will have an opposite phase from CH<sub>2</sub> 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). 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  $^1\text{H}$  and  $^{13}\text{C}$  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. 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  $^1\text{H}$  and  $^{13}\text{C}$  experiments, respectively. NOTE: Due to a bug, you may have to go in and change the offset value multiple times.

5. 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  $^1\text{H}$  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  $^1\text{H}$  and  $^{13}\text{C}$  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 24/256 steps in the indirect dimension. Increase the # of points to 96 and you may be able to stop your experiment sooner.

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