

# HMBC - Quick Reference for ZEUS/ARTEMIS/HADES

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

*NOTE: HMBC picks up long range H-C coupling. This experiment is optimized to pick up <sup>2-3</sup>C-H couplings of 8 Hz, and to selectively remove one bond C-H couplings around 145Hz. Contact Russ @33069 if you have any questions.*

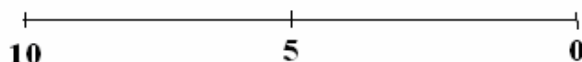
1. *If you already have a <sup>1</sup>H spectrum and know your sw and o1p, you can skip to step 2.* Setup and obtain a 1-D proton spectrum. Be sure to check the tuning and matching of the probe – see instructions in this binder. Determine the optimal spectral window around your peaks of interest allowing for ~ 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 <sup>13</sup>C spectrum for your sample, you should select an **sw** and **o1p** that will include all the carbons allowing for ~10ppm at both extremes. Example: Your <sup>13</sup>C 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). You do not need to reacquire the <sup>13</sup>C spectrum with the new sw. REMEMBER- HMBC spectra will show cross-peaks to quaternary carbons, so do not use the sw from a DEPT experiment or you may miss cross-peaks to quaternary carbons. If you do not yet have a <sup>13</sup>C spectrum, you can either acquire one now or after you run the HMBC, but you will not be able to optimize the **sw** and **o1p** for the <sup>13</sup>C dimension (use the default values unless you can predict the limits from your structure).
3. Type **edc** [enter] and change the experiment number to **2**. Type **rpar** [enter] and select **HMBCGP** [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 <sup>1</sup>H and <sup>13</sup>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**. **Tune and match the probe** (see instructions in this binder). Type **rga** [enter]. Type **zg** [enter] to begin the experiment. NOTE: If you are looking for long range couplings 3-4 bonds, you may need to run multiple experiments with different values for CNST13 (default is 8Hz). NOTE: In some instances, 3 bond coupling is greater than 2 bond coupling.
4. Type **xfb** [enter] to process the 2-D data any time during the acquisition. 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. If you do not see any meaningful correlations at all, go back and double check your o1p values in the **eda** menu.

- There is often incomplete suppression of 1-bond couplings and this will result in cross peaks in the 1H dimension that do not line up with any protons but will precisely bisect the resonance in question (if you overlay the HMBC with the HSQC spectrum it should be clear which peaks have not filtered out completely).

### 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			
TDef =	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