

Instructions for Magnitude HETCOR Spectra on the Varian

Please note:

- Under no circumstances move the magnet or the automatic sampler table.
- This guide assumes that the user is familiar with normal Varian operation as outlined in the instruction manual.
- Unless specifically instructed, do not attempt to use the auto sampler robot
- The new Varian Vnmr software enables the user to easily access and change functions, options, and their properties. If you are not sure about a option, button or command ask the NMR staff for assistance.
- If you encounter problems at any time during locking, shimming, acquiring you should halt the experiment and reload the standard parameters for your experiment.
- In order for shimming to work well, samples should be filtered and prepared in good-quality tubes. Sample volume should be about 0.7 mL. Do not use more than 0.1% TMS.

Conventions in this guide:

- Buttons are depicted with small caps, e.g. **ABORT ACQ**.
- Commands entered in the command window are described in bold letters, e.g. **h1cdcl**, and executed by hitting the return key.

Important basic functions:

- **ABORT ACQ** button -> aborts/halts aquisition
- **CANCEL COMMAND** button -> aborts/cancels last sent command
- **RESIZE** button -> enlarges/reduces spectrum window
- **FLIP** button -> flips the spectrum window over the display window and vice versa
- **jexp_x** command -> switches to experiment number x (1, 2, 3, 4, etc.)
- **ds** command -> switches back to interactive display mode

HETCOR data acquisition:

- acquire a ^1H spectrum in experiment 1 (**jexp1**) using the standard parameters
- phase the ^1H spectrum, then expand the spectrum such that the peaks of interest are displayed on the screen
- type **movesw** to set up an optimized acquisition window for your sample
- re-acquire the spectrum (**ga**), phase and display it. Check that it looks correct
- acquire a ^{13}C spectrum in experiment 2. If time permits, expand, optimize, and re-run the ^{13}C spectrum (note that if your spectrum has downfield quaternary carbons these do not need to be included in the expanded region)
- move the ^{13}C fid from experiment 2 to experiment 3 with **mf(2,3)**
- go to experiment 3 (**jexp3**), do **wft** to check that the ^{13}C spectrum appears correctly, then type **hetcorjsh(1)** to set up a magnitude-mode heteronuclear correlation spectrum
- check that the **nt** and **d1** values are appropriate for your sample (usually the defaults are fine)
- type **go** to start the acquisition - DO NOT USE **ga**!

HETCOR data processing and plotting:

- type **wft2d** to carry out a 2D Fourier transform and to display the 2D spectrum
- click the **MAIN MENU** button above the spectrum window, then click the **DISPLAY, SIZE, FULL SCREEN** buttons
- place the mouse pointer on the 2D spectrum and click the middle mouse button to adjust the vertical scale of the 2D display. Clicking on the baseplane (black) areas will increase the vertical scale, clicking on peaks (colored areas) will reduce it. Alternatively, place the mouse pointer on the multi-colored scale to the right of the 2D spectrum display, and click the middle mouse button to alter the vertical scaling
- type **plhxcor(8,1.5,1,2)** to plot the 2D spectrum along with the 1D spectra along the axes
- expansions are done as for 1D spectra, with the left and right mouse buttons and the **EXPAND** menu button

Finishing up:

- go back to experiment 1 (**jexp1**) and restart sample spinning
- proceed with other spectra or finish up as described in the other instruction book