

Varian HETCOR Quick Help

The HETCOR experiment is available on all the Varian spectrometers.

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HETeronuclear Chemisal Shift CORrelation

The HETCOR experiment is also known as C, H correlation by polarization transfer. The HETCOR is a directly detected 2D ^{13}C , ^1H correlation experiment where cross peaks yield information about the connectivity of protons with ^{13}C nuclei over a single bond. The experiment takes advantage of the large one-bond heteronuclear J coupling ($J_{\text{HX}} \cong 100$ to 200Hz) for polarization transfer. The experiment can be modified to give coupling information over two-, three-, and four-bonds where the J_{HX} coupling constant is typically on the order of 5 to 20Hz.

Please consider what information you wish to glean by performing a HETCOR. You may be better served by considering one of the indirect experiments such as HMQC, HSQC, or HMBC.

Please note that all **bold print** is input at the prompt

- Log into the NMR, start VNMR and join experiment #1 (type **jexp1**)
- Load the desired ^1H parameters and type **su**
- Set the cooling gas preconditioner temperature to 15° C (if you are going to regulate at 25° C, then you can leave the preconditioner at 20° C). Make sure the VT gas is flowing between 10 to 15 lpm and set the temperature to 20° C by typing **temp=20 su**
- Open acqi window, insert the sample, lock, and shim as usual. As with most 2D NMR experiments, do not spin the sample. Adjust the z, z2, x, y, xz, yz, and xy shims.
- After shimming, turn off the lock and carefully readjust (i.e. use the ± 1 button) the z0 (the field offset) to get as close to on-resonance as possible. Turn the lock back on and adjust the lock phase to maximize the signal. Repeat lock off, z0 adjust, lock on, lock phase adjust as necessary.
- Close the acqi window and tune BOTH the ^1H (channel #1) and ^{13}C (channel #2) channel of the probe (channel #1).
- As with many 2D experiments, calibrated pulses are critical for a successful experiment. The HETCOR experiment is especially sensitive to pulse imperfections. At this point you should consider calibrating the 90° pulse widths. If you haven't done this before download our "How to calibrate a 90° pulse" handout or ask a staff member for assistance.
- Collect and process a 1D proton by typing **ga**. Type **ds** (display spectrum) and position the cursors around all the peaks (leaving about 1 ppm on each side of the spectrum) and type **movesw** (move spectral window according to cursors). Note that you should select a sweep width (sw) > 10 ppm.
- Turn autogain off by typing **gain='y'** and recollect the 1D proton. Phase the spectrum. This will serve as the ^1H reference spectrum for the HETCOR.
- Join experiment #2 (type **jexp2**) and load the desired ^{13}C parameters then type **su**.
- Once the experiment has run long enough to determine the desired sweep width type **aa** (abort acquisition). Use the cursors to select the desired sweep width then type **movesw ga**. Phase the spectrum. This spectrum will be used as the reference for the ^{13}C dimension of the HETCOR.
- Type **mp(2,3) jexp3** (move parameters from experiment #2 to experiment #3 and join experiment #3).
- Type **HETCOR(exp #)** (all capitals, so with the above example type **HETCOR(1)**) to convert the ^{13}C parameters to the HETCOR parameters. Type **time** to display the estimated experimental time.
- If the experimental time is too long and you have a reasonably concentrated sample, type **nt=16 ni=64 np=1024 fn=1024 fn1=256 d1=1**. Otherwise the standard parameters should work just fine.
- Type **time**. The experiment completion time will be displayed.

- If you wish to gain some information about the longer range correlations type **hmult='y'** (default is **hmult='n'**). If you really want this information you should probably be running an HMBC.
- Type **au** (submit experiment to acquisition and process data) to start the run. Do not be concerned if an ADC Overflow message flashes. The experiment will not be adversely affected.
- When completed type **wft2d** (weight and Fourier transform 2D data)
- You can readjust the vertical display by typing **vs2d=vs2d*1.2 dconi** to increase the vertical scale or **vs2d=vs2d*0.8 dconi** to reduce; there are also buttons that will increase and decrease the vertical scale of the 2D spectrum by 20%; the parameter **th** can also be adjusted (0 shows the most noise, higher values increase the cutoff threshold), the color scale on the right of the spectrum will show you the value of **th**; clicking with the middle mouse button on the color scale on the right of the 2D spectrum allows you to interactively adjust **th**.
- If the peaks look to be poorly phased (as you traverse the peaks along a row there will be a stripe that is yellow/orange on one-side and blue/purple on the other side), then you will have to phase by hand. See the "Phasing a 2D" handout for this procedure available on our website.
- To plot your data interactively, type **dconi** and use the VNMR menu buttons. Once you have processed the data to your liking you can also use the plotting command **plhcor**. For example if your ¹H 1D is in experiment #1 and your ¹³C 1D is in experiment #2 type **plhcor('pos',30,1.5,1,2)**. For more details on plotting 2D spectra refer to the "Plotting a 2D" handout available on our website.
- Save the 2D data set with the **svf** command.
- Turn VT regulation off by typing **temp='n' su** (you may need to go in several steps to get from whatever temperature your sample is at back to 20° C – DO NOT CHANGE THE TEMPERATURE BY MORE THAN 10 DEGREES EVERY FIVE MINUTES). Set the preconditioner back to 20° C.