BARE BONES GUIDE TO gNOESY w/ ZQF filter on the Inova-400&500 in VNMRJ 3.2

This guide is written assuming proficiency in basic operation of the Varian NMR instrument. You should be experienced in performing basic 1-dimensional NMR experiments before attempting to perform 2D experiments on your own. Please ask for help the first time you perform this, to minimize your frustration.

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Important Considerations before starting:

- You can perform this experiment in either the 4-nucleus (n4n) probe, or the triple-inverse (ntr) probe; however, the sensitivity is MUCH better with the ntr probe. It is not a big deal (~5 minutes) to change probes, so unless you have a concentrated sample, it is suggested that the ntr probe be used. NOESY cross-peaks can be very weak, so good S:N can be important.
- NOESY, when done properly, is a time-consuming experiment (due to relaxation considerations). Plan for at least 2-hours, even for relatively concentrated samples..

1) Make sure you are working in Exp:1 (type **jexp1**, and verify you are working in Exp:1).
2) Acquire a normal ^{1}H NMR spectrum, and save it to be used as a 1D-Trace for the 2D-plots.
3) *Type* **mf(1,2) <space> jexp2 <Enter>** (this means: "move fid" … meaning, "copy" your FID and parameters from Exp:1 to Exp:2, then join Exp:2) – *Note, that you could substitute "3" for "2" in the above line if you have already setup another experiment in Exp:2… i.e., mf(1,3) jexp3 … would copy the 1H NMR located in Exp:1 to Exp:3, and then join Exp:3 for further setup.*
4) Place the red-cursors around the region of interest – DO NOT ZOOM IN. (Note: if you select a region with peaks outside that region, you will have *folded* peaks in your spectrum...this can be OK, or it can be a problem; depending upon where the folded peaks land).
5) Type **movesw** , (or *click* on [MoveSW] in the [Acquire] panel. and this will set the spectral width and offset to match your selected window – ignore the apparent change to the shift scale at this point – go to the next step.
6) Go to the [Experiments] Pull-Down menu, via [*Convert Current Experiment to Do…*], [Homonuclear Correlations], [NOESY].
7) In the [Acquire]/[Default] menu/panel you will find "Acquisition Options"… this is the easiest way to setup the NOESY experiment:
   a) The default "Scans per t1 increment" (nt) is 16, which gives the cleanest, most artifact free NOESY spectrum. You can reduce this to 8 and still get reasonably good data with strong/concentrated samples. 4 scans is not recommended, regardless of sample concentration.
   b) "t1 increments" (default is 200) can be reduced to 128 to save time, and with linear prediction can still yield excellent data.
   c) Relaxation Delay, default is 1 sec, is usually OK; however, a longer delay can greatly improve the data for many samples (if you have the instrument time available).
   d) NOESY Mixing Time: This really depends on your sample, and choosing the optimum requires an understanding of cross-relaxation processes and competing relaxation mechanisms. 200ms (default) is pretty short, so recommended values would be 400-600ms as good starting values for most small molecules (in the extreme narrowing regime). If you don't understand that last part, you should study Chapter 8 of Tim Claridge's book *High-Resolution NMR Techniques in Organic Chemistry*.
8) Type **time** (or *click* on [Show Time]) to see how long the experiment will take (*optional*).
9) *Click* on [Acquire] **(**or type **cpgo**) to start the experiment.
   a) You can process the data while it is acquiring via the [Process][Default] panel.
10) Save the data using "Save As…", navigate to your Directory in /nmrdata/rgroups/group/user, the same as you would with a 1D spectrum. Verify that the data is saved properly.
11) Type **jexp1** , eject your sample, and re-insert the reference. Lock on the reference as usual.

Processing in MestReNova: Be aware this is Phase-Sensitive data, and you should pay attention to the First Point multiplyer (0.5) under the Apodization panel to minimize noise streaks in both dimensions. Use Gaussian or Cosine-squared apodization, and use care in phasing and baseline correction to get the highest quality result. NOESY is one of the most difficult experiments to process optimally, so don't be shy about asking for help the first few times.