

## 2D COSY and DQCOSY (VnmrJ ChemPack)

IU NMR Facility – September, 2010

### *Summary:*

COSY, DQCOSY and the gradient versions gCOSY and gDQCOSY are easy to run and amongst the most useful 2D NMR experiments. They give correlation cross peaks for direct scalar coupled atoms (homonuclear). COSY experiments using nuclei other than proton, such as  $^{19}\text{F}$ , are also possible.

COSY is typically run in absolute value mode. The gradient version is preferred when it's available due to cleaner results and the possibility of using as few as one transient per 2D increment ( $nt=1$ ) allowing for 2D spectra to be acquired in minutes or less.

DQCOSY provides the COSY information with singlets suppressed, an inphase diagonal so that correlation peaks close to the diagonal are more easily seen and no COSY relay peaks, but at lower S/N than COSY. The phase sensitive DQCOSY can give high enough resolution to extract coupling constants. The gradient version gDQCOSY is especially attractive as it is free from subtraction errors and can also be run with  $nt=1$  to provide fast results. DQCOSY can be run in absolute value mode but is phase sensitive by default ( $phase=1,2$ ).

### *Experiment Procedure:*

- 1) Optional: Lock, shim, setup a 1D proton experiment, choose solvent, acquire a quick 1D proton spectrum, reference and save it. This step is helpful but not required. You can also optionally determine  $pw90$  for your sample to get best results. See the VnmrJ 2D Guide for this procedure and a general overview.
- 2) Type "**iucosy**" or "**iudqcosy**" to setup up an optimized experiment for that spectrometer. Or using the VnmrJ "Experiments" menu item at the top, choose "Setup NEW parameters to do ....." -> "1H – 1H Hom. Correlation Experiments" -> "COSY", "Gradient COSY", "DQCOSY" or "gradient DQCOSY". Make. sure the solvent is correct and sample spinning is off.
- 3) For longer or shorter experiments vary **ni**, **nt** and **d1**. **nt** should be a multiple of 8 for COSY or DQCOSY but can be 1 for gCOSY or gDQCOSY. A "fast" gCOSY or gDQCOSY can be obtained with **nt**=1, **d1**=0.2 and **ni**=96 for example. The "Acquire" -> "Default" template contains **nt** and **ni** and the experiment duration can quickly be checked with the yellow "Show Time" button (from "Acquire" tab). Generally increase  $nt$  if you have a small amount of sample for better S/N, but otherwise increase  $ni$  for better S/N and resolution in the second dimension.

- 4) After acquiring the data, save it. Processing can be done using the VnmrJ "Process" -> "Basic" template. If **ni** was changed from the default you might have to turn linear prediction off from this template (check box) or type "**lp2d**" to reset it. Linear prediction can also be reset from the "Process" -> "Default" template using the "Auto LP F1" button. You can also simply use the green "Autoprocess" button (from "Acquire" tab) if the default experiment was run.
- 5) Use the "Process" -> "Default" template for referencing and optional baseline correction. Use the 2D toolbar on the upper right side to adjust the display.
- 6) Use the VnmrJ "Process" -> "Plot" template for plotting or type "**plot2d**" to use the IU macro for plotting that allows you to plot the 1D proton spectrum along the edges of the 2D spectrum, with the 1D spectrum stored in another experiment.
- 7) If you like the results, you can save the data again, overwriting the original, to save your modified processing parameters with the data.

*Notes:*

By default we have COSY set to absolute value mode and DQCOSY set to phase sensitive mode. Phase sensitive COSY and gCOSY are rarely used and require separate pulse sequences.

To convert to phase sensitive type: "**phase=1,2 f1coef = '1 0 0 0 0 0 -1 0' pmode='full' ph ph1**"

For absolute value type: "**phase=1 pmode='partial' f1coef='1 0 0 1' av av1**".

"Fast" versions of AV or phase sensitive gCOSY or gDQCOSY can be run with nt=1 and d1=0.1.

The first few increments of a DQCOSY experiment may have little to no signal, this is normal.

More than most 2D experiments, DQCOSY benefits from a higher **ni** value. If you have extra time to obtain better data, consider increasing ni rather than another parameter.

DQCOSY (non-gradient) is currently not available as the VnmrJ version without gradients does not work.