

2-D COSY/gCOSY

IU NMR Facility – August 31, 2004

Summary:

COSY and its gradient version gCOSY are some of the easiest to run and most useful 2-D NMR experiments. They give cross peaks for scalar coupled (through bond) ¹H atoms. The experiment will be run in absolute value mode for the best possible signal to noise. gCOSY can be run with *nt* = 1 allowing for experiments as short as just a few minutes.

To get improved resolution, a reduced diagonal and to be able to extract coupling constants run DQCOSY or ECOSY variants instead.

Experiment Overview:

- 1) Set up is similar to other 2-D homonuclear experiments.
- 2) Acquire a ¹H spectrum with a 90 degree pulse width, phase and reference it. Optionally copy the parameters to another experiment.
- 3) Type **iuco**sy or **iugco**sy or use the Other/2D menu and select **co**sy or **gc**osy. For a longer or shorter experiment vary either **ni**, **nt** or both. **nt** should be a multiple of 8 for **co**sy but can be 1 for **gc**osy. Type **go** or **ga** to begin the experiment.
- 4) After acquiring the data, process with any of these options: 1) Type **iucoyproc** or use the Other/2D menu under Processing and select **co**sy – both are the same. 2) Use the general 2-D processing command **wft2d**.

Display 2-D data with **dconi**.

By default COSY and gCOSY are obtained in absolute value mode. It can also be run in phase sensitive mode for better resolution with **phase=1,2** and using **wft2da** to process the data. However DQCOSY, ECOSY or gDQCOSY are better phase sensitive options.

Procedure:

- 1) Set up ¹H experiment as normal in EXP1. Verify that *pw* = 90 degree pulse. This is a critical parameter and will typically be 20-25 μs for quad and broadband probes and 8-10 μs for proton detection probes.
- 2) Lock, shim and obtain ¹H spectrum as normal except DO NOT SPIN THE SAMPLE. As the sample will not be spun it is usually worth checking the non-spin shims (x,y, etc.) also.
- 3) Set the cursors just outside either ends of the spectrum and type **movesw**. This command adjusts **sw** and **tof** so that only the region between the cursors will be observed.
- 4) Obtain another ¹H spectrum with the new **sw** and **tof**. Autogain is not allowed with 2-D experiments. A good way to set the gain is obtain a 1-D ¹H spectrum with *gain='n'* (autogain on), then type **gain='y'**.

- 5) It is convenient to keep the 1-D spectrum in EXP1 and run the 2-D experiment in EXP2. To do this type **mp(1,2) jexp2** to transfer the 1-D info then set up the cosy experiment by typing **iucosy** or use the Other/2D menu under Setup and select **cosy** to run the same macro.
- 6) With the default parameters for cosy of **nt=8 ni=96** and **d1=1** the experiment will take 15 minutes.

For a quick check of the experiment type **phase=1** and **ni=1** to obtain just the first increment of the 2-D experiment. It should look much like the 1-D ¹H spectrum.

- 7) Type **ga** to begin the experiment.

Processing:

- 1) Type **iucosyproc** or use the Other/2D menu under processing and select **cosy**. You can do the same by typing **fn=2k fn1=2k sinebell lp2d(512) wft2d**. You may have to make sure no other window functions are active (i.e. set **lb='n'**). **lp2d** is a macro that sets up linear prediction and a window function for the F1 dimension. For the **iucosyproc** macro you will usually want to answer **y** to use linear prediction. Use the same procedure for **gcpsy**.
- 2) If the 2-D spectrum appears weak or not at all (black screen) right click anywhere in the spectrum to increase the 2-D vertical scale (**vs2d**). Once you can see peaks you will usually want to right click on a medium strength crosspeak to set **vs2d**, or you can always adjust it manually. There are menu options to adjust **vs2d** as well and most other 2-D display options can be found in the display menus.
- 3) The macros **plot2d** or **iuplcosy** can be used to plot the 2-D data with the 1-D spectrum on the sides. Type **plot2d** and follow the prompts. Typical inputs for **iuplcosy** are something like **iuplcosy(12,1.4,1,3,3) iupage** which will plot up to 12 positive contour levels (1 negative level by default) with a spacing of 1.4 and plot the spectrum in EXP1 on the sides with a scaling factor of 3. Learn more about either with **man('cosy')** or **man('plot2d')** help files.

Notes:

It is often useful to save your data again after processing (overwrite the original data) to save the processing parameters so that the next time you call up the data you can simply type **wft2d** to view it.

If you need to use solvent presaturation (with samples in D₂O) for example, it is best to establish parameters in a 1-D experiment and use the **man('cosy')** help file.

It is sometime useful to view the absolute value 2-D spectrum if you acquired a phase sensitive one. Type **av av1 dconi** to do this. To return to the phase sensitive display type **ph ph1 dconi**.