

## Lab# 4 Problem Set:

**This problem set is due on Lab #5.** As usual, if you have any problem getting time on DRX300, logging-in, finding the sample, collecting data, processing data, analyzing data, or plotting data, please see either Mark Girvin or Sean Cahill. *Late reports will be marked 50% off.*

- Determine the  $^1\text{H}$  90 degree pulse and carrier frequency for the peptide on the DRX300. What are the values?
- Collect and plot out a 1D  $^1\text{H}$  spectrum of the peptide on the DRX300 using water suppression.
- Collect a 2D TOCSY spectrum of the peptide on the DRX300 using water suppression. Plot out the “amide tower” region from 8.8 – 7.7 ppm in F2 and 8.8 – 0 ppm in F1.
- Assign the spin systems that are marked on the attached 2D DQFCOSY spectrum of the peptide. Refer to the primary sequence given in this handout. You can mark the spin system directly on the attached plot (for example, write “gly” if the crosspeak is consistent with a glycine residue). If you cannot determine a unique residue for a given crosspeak, write down all the residues that are consistent for the spin system. Indicate all  $^1\text{H}$  chemical shifts belonging to each spin system in order starting with HN.

You may use the NMRView analysis software to make assignments. NMRView datasets are located in `nmr0:/unmr0/data/class/NMRVIEW` (dataset naming is based on the experiment that was used). To view the datasets from your account, do the following (also see instructions in NMRView handout):

- log into nmr0
- type “`cd /unmr0/data/class/NMRVIEW`”
- type “`nmrview`”
- open up a dataset by clicking under the main menu on “Dataset”, click on “Open and Draw Dataset” then click on the desired dataset and click “Open” in the File menu. Multiple files can be opened by

clicking on the dataset name and click “Open” in the File menu. Click on “Close” in the File menu when all datasets have been opened.

- Use the  $H\alpha$  chemical shifts of the spin system assignments that you can uniquely identify to predict the 2<sup>o</sup> structure of the peptide – refer to table of random coil values of  $H\alpha$  chemical shift given in the lecture handout (hint: residues in  $\alpha$ -helical segments will have  $H\alpha$  shifts  $\sim 0.3$  ppm upfield of random coil value and residues in extended strands like  $\beta$ -sheets will have  $H\alpha$  shifts  $\sim 0.3$  ppm downfield of random coil value).