Names _____

Chem 453, Fall 2012
Experiment 4 - Worksheet

Each pair of students should work together to fill out their own group worksheet. You are welcome to discuss your results with members of other groups, but each group should hand in their own results, not a replica of some other group's results. The worksheet is due to me by 4 pm on the same day as your scheduled Final Exam (18. Dec. for Section 002 and 21. Dec. for Section 001). I will be available during the scheduled Final Exam time on those two days (1:00pm - 2:50pm) to answer any last minute questions you may have.

Dipeptide Results: (Name of peptide _____)

1. Based on the exact mass of your assigned dipeptide to five significant figures, what are the possible options for its amino acid composition?_____

    a. What is the predicted exact mass of the *uncharged* species in Da? _____

    b. What is the observed exact mass of the *uncharged* species in Da? _____

    c. What is the relative error for your observed mass in ppm (parts per million)? _____

2. Based on your NMR evidence, draw the expanded chemical structure for your dipeptide. Next to each proton in the chemical structure, indicate its NMR resonance assignment in ppm.

A-Ω Oligoeptide Results:

1. Based on the LC/MS results, what is the exact mass of the uncharged species of the A-Ω oligopeptide ?. _____

2. Based on this mass, predict the number of amino acid residues contained in the A-Ω oligopeptide_____

3. Based on the NMR evidence and the exact mass, predict the amino acid *composition* (not sequence) of the A-Ω oligopeptide. Use a sentence or two to describe your evidence for each claim. This evidence can come either from the spin systems you observe in the NMR spectra, or mass differences in the observed and calculated mass spectra. You may not need the entire table below to do this.

Amino Acid Residue	Evidence

Amino Acid Residue	Evidence

4. Report your predicted sequence for the A-Ω oligopeptide.

   a. Using the 3-letter amino acid labels, indicate your predicted amino acid sequence for the A-Ω oligo peptide. This is an example of the format that you should use (recall that the C-terminus is an amide):

   $$^{+}H_3N\text{-Ala} \rightarrow \text{Leu} \rightarrow \text{Gly} \rightarrow \text{Ser} \rightarrow \text{Tyr} \rightarrow \text{Asn} \rightarrow \text{Thr-}NH_2$$

   If you encounter resonances that overlap one another in your NMR spectra, you may discover that it gives rise to some uncertainty in your sequence assignment. If this is the case, then you should indicate this in your sequence diagram by showing a branch point.

b. Describe the steps that you used to arrive at your proposed sequence(s).

c. Attach a copy of your resonance assignments. This can be done from within *Sparky* by selecting Peaks > Resonances > Resonance List (rl) to display the list and then click the "Save" button to save the list as a text file, which can be included with this worksheet.

5. Mass spectrum results for the A-Ω oligopeptide:

   a. What is the predicted exact mass of the *uncharged* species in Da? _____

   b. What is the observed exact mass of the *uncharged* species in Da? _____

   c. What is the relative error in ppm (parts per million) for your observed mass?

      _____

6. Two of the 2002 recipients of the Nobel Prize in Chemistry, John Fenn and Kurt Wüthrich, made scientific contributions, which helped you to characterize your dipeptide and the Snow White oligopeptide in this assignment. Briefly describe these contributions.