1D/2D NMR Project for Chem 232:
DUE At 4:00 PM December 13, in box outside my office (Room #6, Stauffer I)

The assignment is to acquire, process, assign, and interpret the following spectra and then determine the two-dimensional, chemical structure of several separate unknown molecules. You should turn in a drawing of what the unknown molecules are, an assignment table of $^1$H/$^1$H/$^1$C/$^1$N/$^1$F chemical shifts or chemical shift assignments on the 2D drawing of the molecules (any format correlating the assignments to the structure is fine). All observed resonances should be assigned. Although not necessary, assignment of the observed peaks on prints of the HSQC spectra is helpful.

**Week 1:**
1) 1D $^1$H, 1D $^{19}$F, 1D $^{13}$C

2) Measure 1H T1 relaxation times for mixture

**Week 2:**
3) $^1$H/$^{13}$C HSQC with multiplicity editing

4) Sensitivity-enhanced $^1$H/$^{13}$C HSQC

5) $^1$H/$^{13}$C HMBC

6) $^1$H/$^{13}$C H2BC

7) COSY

8) TOCSY to identify resonances connected by ~5 magnetization transfers

9) ROESY [standard, many ROEs]

**Week 3:**
10) $^1$H/$^{15}$N HSQC

11) $^1$H/$^{15}$N HMBC

12) TOCSY to identify resonances connected by only ~2 magnetization transfers

13) ROESY [to observe crosspeaks only between $^1$H very close in space]

14) Experiment to measure JHH coupling constant
The first week is an overnight on the 500 (~setup time of 1 hour).

The next 2 weeks are overnight experiments on the 600 (~setup time 1\textsuperscript{st} week of 1.0-1.5 hours, 2\textsuperscript{nd} week of 0.5-1.0 hours).

The data are to be acquired in groups, for the 3 weeks. Data processing time can be arranged at any time, contact me to arrange times.

The sample is in DMSO. There are several different unknown molecules in the sample, with approximately the same concentration of each, ~5-10 mgs total (between them), a total Molecular Weight of ~1500 between them (within ~30\% of 1500 MW). The only element in the unknowns that you will not either directly or indirectly detect in the course of the various experiments is Oxygen- there are at least 10 Oxygen atoms. There are a number of exchangeable $^1$H (attached to N or O). Some of these are readily observable, but a few of them are hard to observe, and may not be observed in the 2Ds. For now, that is all of the information that I am giving you, but as you start going through your data, I can help you with a little more information. To answer a few other questions ahead of time: No, you cannot run an HPLC or Mass Spec on the sample.

There is only one sample to be shared by the 3 groups. It is preferred that when trying to interpret the data, you stay within your groups. If all people within a group contribute, you can turn in just 1 report for each group. If not, you should turn in a report individually (or in some smaller group). You need to draw structures for the molecules that you identify with $^1$H, $^{13}$C, $^{15}$N, and $^{19}$F resonance assignments on the structure or in table that can be correlated to the drawn structures. You do need to stereospecifically assign resonances where possible. When you are far enough along in the project, you can ask more specific questions on what to turn in.