59-542: NUTS Assignment **Fall 2008 Term**

In this assignment, you must process the supplied raw data files using NUTS, and prepare them as publication quality spectra for a paper or thesis (using Powerpoint or whatever software you prefer to use). The final figures should include a title and description of the processing that has taken place. The best command for pasting to Powerpoint is to "Copy Standard Metafile to Clipboard", which is accomplished by Alt-Shift-C (at this point, select portrait or landscape).

The raw data are contained within the zip file **nmrassign.zip** which is available for download from the website.

The files are as follows:
Eb1H.fid
Ebc13.fid
alleve.002.fid
$glycine.2.fid
$glycine.4.fid
conh35cl/

The due date for this assignment is anytime before the final examination, and it counts for up to 5 additional bonus marks towards your grade.

Processing Instructions:

If you follow the instructions closely, you will easily get the hang of this. All data should be baseline corrected prior to doing anything else. The command BC baseline corrects the FID to eliminate spurious DC offset effects! As well, use the zoom command (double click left button) to expand regions of interest in the spectra.

1. Ebc13.fid
This is a carbon-13 NMR spectrum of ethylbenzene acquired at 8.46 T using a Varian NMR spectrometer. Perform the following processing:

LB - set the appropriate line broadening in Hz (this may take a few tries to get it right)
EM - exponential multiplication (this will test your applied line broadening)
FT - Fourier transform
PH - phasing - set the pivot first by holding down the left mouse button and pressing "P" where you want the pivot. Zeroth order phasing is accomplished by holding down the left mouse button, first-order with the right mouse button.
PP - peak picking - the minimum threshold for peak picking can be set with the command MH (minimum height). Ctrl-P can be used to toggle the display of peaks off and on.

2. Eb1H.fid
This is the proton NMR spectrum of the compound above at 7.05 T. After performing LB and EM, you will notice that the resolution of peaks in this spectrum is quite poor. Therefore, you must ZF (zero fill) once or twice to improve the appearance of the proton multiplets. There is

also a solvent peak in this spectrum. Identify it in your final spectrum.

You will be plotting two different spectra here: the full proton spectrum and then an expansion of the aromatic region (this will show that the resolution enhancement has been done properly.
ZF - zero filling is necessary
PH - if necessary
On the full proton spectrum:
ID - integrate display - use this to integrate and set the relative values of the peaks. Do not integrate the solvent peak.
On the expanded aromatic region:
PP - peak pick to show all of the individual resonances.

3. alleve.002.fid
This is a ^{1}H NMR spectrum of the pain-killer alleve dissolved in D2O at 8.46 T.
You will be plotting two different spectra here: the full proton spectrum, and an expansion of the aromatic region (ca. 7.0 to 8.0 ppm). Notice that the peaks in the aromatic region are not well defined, so we will apply window functions to improve the resolution.

Process the full proton spectrum as usual, and perform a peak pick (PP). Identify the TMS peak in your final spectrum.

To get a resolution enhanced spectrum, set LB = -0.5 and GF = 0.6, and perform EM as usual. You will have to reprocess the spectrum to do this. Expand the aromatic region and you will see that the S/N is worse, but the peak positions are clearly defined. Zoom in on the aromatic region, perform a peak pick, and plot this spectrum as well. Ideally, it would be nice to have a smaller aromatic region as an inset above the full proton spectrum.

4. $glycine.2.fid & $glycine.4.fid
These are solid state carbon-13 CPMAS NMR spectra of glycine acquired at 11.7 T on a Bruker NMR spectrometer. You will be processing two spectra here, and plotting them on the same spectrum using a dual display feature in NUTS.

You will notice that the FIDs look strange because of a string of points at the beginning. This is the result of a digital filter that is applied on Bruker spectrometers to cut off any unwanted signals outside of the desired spectral window. Process each of them as follows:

BC - baseline correct
RD - rotate data - this eliminates the digital filter at the front of the FID
EM - try an LB of 30 Hz or so.
FT
PH - phase the spectra.

Save each spectrum with the suffix ".nmr"
To compare these spectra:
Read in $glycine.2.nmr. Type **AL** (this loads the spectrum into the dual display buffer).
Read in $glycine.4.nmr. Type **AS** (this gives dual display). You will be able to easily pick the

isotropic peaks now.
After you have processed spectra, you will see that they are slightly different - they were acquired at two different magic-angle spinning speeds, so the spinning sidebands move around. Identify the isotropic centrebands, leave the sidebands unmarked. While both are still displayed on the screen, use Alt Shift C to copy them to Powerpoint. You will only have the one file with both spectra displayed.

5. conh35cl
This is a raw Bruker dataset containing a cobalt-59 NMR spectrum of Co(NH3)5Cl acquired at 11.7 T, with very poor signal to noise. Processing raw data is slightly different. Bruker data files have a directory format, with the filename at the top, and each individual experiment contained within a numbered directory. To import this file into NUTS, open the file:

conh35cl/1/fid

it will auto-detect and import the file properly.

This spectrum has very poor S/N, and will require a lot of signal enhancement. Process as follows:
BC, RD, LB = 1000 Hz (yes! 1000 Hz), EM, FT

We are interested in obtaining an accurate linewidth at half height for this peak. Use the LF command as follows:

Type **LF**. All the necessary commands are in the Fit menu while LF is activated. Click once under the peak, and a simulated lineshape will appear. Type O and wait for the optimization to finish. Type I to see the results. The linewidth should be between 1800-2000 Hz. Hit Enter to exit LF and return to the main menu. Make a plot of this spectrum, and note the linewidth on the plot.

Summary:
The final package should have the following (all spectra should be clearly labeled, with title, etc.)

- Ebc13.fid: One plot, with peak picking
- Eb1H.fid: Two plots: (a) full proton spectrum with integration; (b) expanded aromatic region with peak picking
- alleve.002.fid: Two plots, same page: (a) full proton spectrum with peak picking; (b) expanded resolution enhanced aromatic region with peak picking (try to put these on the same page)
- $glycine.2.fid & $glycine.4.fid: Two spectra, dual display plot: Identify the isotropic centrebands
- conh35cl: One plot: Plot the spectrum, identify the linewidth at half height and chemical shift

Please hand this in before the final examination!