Spectra from the 400MHz NMR for the Perkin lab.

DO NOT put any samples into the system with less than 4cm depth of liquid in the tube, or with significant amounts of solids. Also remember that the minimum tube length is about 6.75 inches (about 170mm) - **any short tubes will be removed**.

Do NOT stick labels to tubes – they can interfere with the sample changer mechanism and with spinning. If they fall off inside the magnet they create major problems. If you write directly on the tubes, only use the top inch or so of the tube.

You **MUST** provide the information requested on the sign-up form for inclusion in the title of each spectrum (unshaded cells). To retrieve your spectrum you require:

1 the spectrometer name (there are 3 spectrometers; **400A**, **400B**, and **400C**),
2 the **date** code for when your sample was **run** (*not* when it was submitted), and
3 the **NMR experiment number**.

Once your sample has been run, this information will have been entered into the sign-up form by whoever ran your sample (shaded cells) - your spectrum cannot be retrieved without this information!

The data is supplied at present by a file server named **chnmr3**. You may use the PCs in the lab to access it from which you will be able to print out a hard copy. Access should also be possible from the departmental clusters, but not using wireless connections or external computers. The data on the fie server can provide a basic printout without special software, or can be further processed by suitable NMR software.

As an example, to fetch data from 400A:

1 Double-click the icon **my computer**.
2 Double-click the network drive **new400s** on chnmr3 (R:)
3 This gives a window allowing choice between the three spectrometers, 400,A, 400B, and 400C. Choose the appropriate spectrometer. – in this case **400A**.
4 This should give a list of users including ug1, ug2, and ug3 - for years 1, 2, and 3. Open the **ug3** folder which contains another folder named **nmr**. Inside this is a list of date codes (e.g. Sep28-2006). Opening a **date folder** shows a folder for each **experiment number**.
5 Double clicking the **experiment number** folder shows various files and the folder **pdata**. Double clicking **pdata** shows a folder named **1**: opening this shows more files including a **file starting with the date code** and the suffix **pdf**.
6 The **pdf** file can be opened with the Acrobat pdf reader program. There is also a file **peak.txt** which can be opened with Wordpad: this contains the list of peaks.

The date, the name of the student, and a sample identifier code will be included in the title field of the pdf file of your spectrum. This is to reduce the chances of plagiarism.

If you have access to an NMR program such as MestRe-C or MestReNova you can use it to look at the files on the network drive directly.

A very basic introduction to using MestReNova software.

This is a new general purpose package for processing NMR data - some features are not as good as they might be as yet. A basic design feature is the copying of some

ideas from Powerpoint to create a set of spectra linked together. This feature can be ignored. For most basic purposes the program can be operated from its main toolbar.

Start by creating a new blank document and choosing open. There seems to be a bug here: sometimes the program takes a minute or two to respond to the open command. Patience!

In the file open window, choose my computer, new400s (drive R:) and then the appropriate spectrometer. Open the user directory (ug3) and the directory nmr – this shows a list of date codes. Double click on the required date code to get a list of experiment numbers and double click the appropriate number. This shows various files and a directory pdata. Open this and the directory inside it to obtain a list of files. Double click the file 1r to get your spectrum.

This should produce your spectrum in the main window. Icons at the right of the main toolbar control vertical size and shift scale: ”manual zoom” allows selection of a specific range in ppm or hz. Some icons such as “zoom in” remain active until clicked again. If all the icons become greyed out, click in the spectrum window.

The other icons likely to be useful are TMS, peak pick, and integral. The first lets the sift of any line be set to a corrected value. Peak pick has various modes but automatic works well. Automatic integration is less good – in manual mode click to the left of a peak and drag across to define an integral. Left click on an integral to drag them all up and down a page, right click for various options.

The multiplet icon sounds clever, but I thought it a bit disappointing. There are a couple of curly arrows in the toolbar which are undo and re-do: very useful.

The only way to alter the maximum size of the integrals is to right click on the spectrum window and go to properties.

Plotting is straightforward. The toolbar icons convert the current spectrum window to a full page plot – alternatively you can select all in the edit window, copy, and paste.

To start work on another spectrum, go back to “create new document”.