Operating Instructions for 1D Spectra
Mercury 200 NMR Spectrometer

May 11, 2011

Remember:

(1) Your NMR tube should contain enough sample to reach a depth of **at least 4 cm**.

(2) A clear solution with no floating particles is required to get a good shimming.

(3) On the 200 MHz instrument always spin the sample.

Setting up the software

1. Login to the computer.
2. Click on the icon to start the VNMR software.
3. To eject the previous sample enter **e**.
4. Place your sample in the spinner, place it on top of the magnet and enter **i**.

5. **IMPORTANT**:

   Enter **ss** (This retrieves the standard shims).

6. *Click on* **Main Menu** of the VNMR software.

   Click on **Setup**

   then *click on* **H1, CDCl3**

   OR

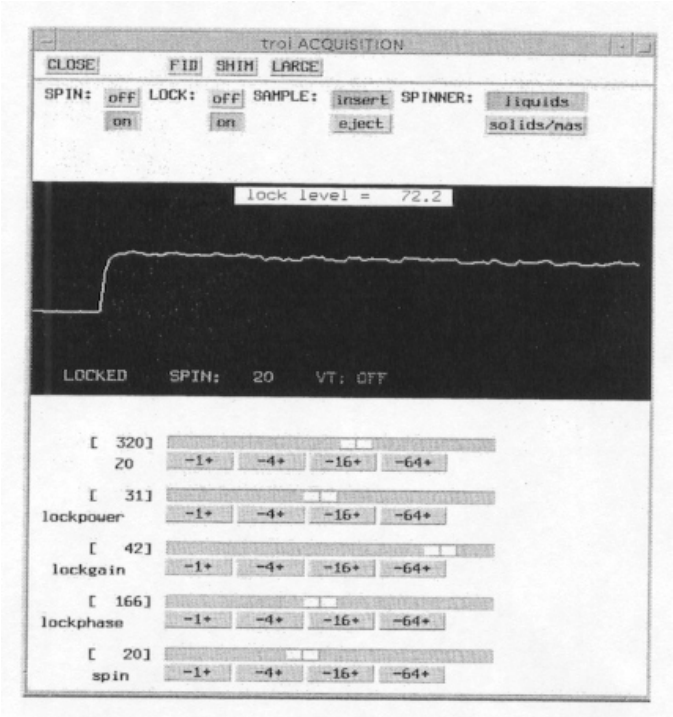
   click on **Nucleus, solvent**
   then specify the nucleus (H1 or P31)
   then specify the solvent

7. Enter **su** .

Locking and shimming the magnet

8. *Click on* **Acqi**

9. In this new window, *Click on* **LOCK** to display the lock screen shown below.



10. Use the sliding bar to set the spin parameter to 15-20.

11. The deuterium lock signal should appear as above. If not, do the following:

    Click on **Lock**: **Off**

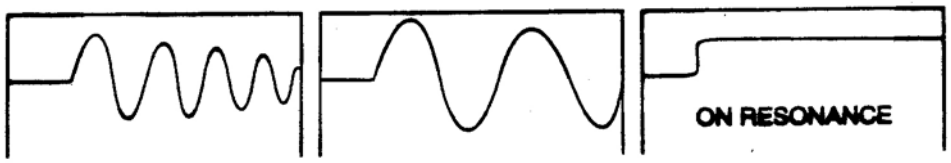
    Set the **lock gain** to the **maximum**, the **lock power** to **30-35** (the lock signal may look something like the left figure below),

    Right or left click repeatedly on the -16+ Z0 button to move in the direction that makes the lock signal look more like the middle figure below,

    Continue until it looks like the figure on the right, then

    Click on **Lock: On**.

    Reduce the lock power and gain until the lock level is on scale (i.e. <100).



Notes: **Before** you start shimming -

- Make sure the lock phase is set correctly. The correct lock phase setting is shown on a note attached to the monitor. The lock phase normally does not need adjusting but sometimes people set it incorrectly.

- The lock power should not be set so high that it causes saturation (large fluctuations of the lock level). The lock power is set correctly if, when you turn down the lock power, the lock level goes down and stays down and does not rebound higher).

[N.B. the solvents acetone, acetonitrile and methanol need quite low values of lock power (10 or less) or saturation will result. Increase the lock gain to compensate.]

Click on **SHIM** to get the shim screen

12. Change Z1C to maximize the lock level. Do this by clicking on the Z1C –1+ button to cause the lock signal to increase. The object is to get the "current lock level" as high as possible. A coloured bar graph is also provided – try to get it as far to the right as possible.

13. Change Z2C to maximize the lock level. Do this by clicking on the Z2C –1+ button to cause the lock signal to increase. Find a new maximum.

    → **If the lock level goes over 100, decrease the lock gain.**

14. Repeat steps 12 and 13 until the best (maximum) lock signal is found.

15. Close the acqi window

16. Enter **ga** (this will start the data acquisition).

Data Processing

17. When the spectrum appears, enter **aph dscale**.

18. To expand the display horizontally: click with the **left** mouse button in the spectrum where you want the left edge of your plot to be and then click with the **right** mouse button where you want the right edge to be, then *click on* Expand in the menu.
Alternatively, you could enter, say, **sp=0p wp=10p** (sp designates the **right edge** of the display and wp designates the **width of the display**).

    To expand the display vertically: place the mouse directly over a peak, press and hold down the **middle** mouse button and raise the peak to the desired height.
    The command **vsadj** will always make the highest peak in the display to be on scale.

19. Optional: To create a text comment for plots enter **text('*whatever text you want*')** .

20. To integrate:

    Enter **ds**

    Enter **cz**

    Click on **Part integral** then *Click on* **resets**

    Using the left mouse button, starting at the left end of the spectrum, Click to the left and right of each peak you want to integrate. Enter **ds** when complete.

    Enter **isadj** , (this adjusts the integral scale so that all are on-scale).

    Enter **dc** . (If integrals are still not flat enter **bc**).

    To normalize, place the cursor on an integral, *click on* **Set Int** and enter the value for the integral.

    To plot, enter **pl pscale ppa pir page**

21. To peak pick:

Enter **ds**

Click on **Th**

Adjust the threshold.

If desired, enter **dpf** to see the chemical shifts on the display.

If too many peaks are listed, enter **ds** , *click on* **Th**, then use the left mouse button to move the threshold higher and enter **dpf** .

To plot spectra with peaks listed, enter **pl pscale ppa ppf page** .

To get a line listing on a separate piece of paper enter **pll page** .

To save your data:

Enter **svf** . Then supply the filename.

To retrieve data:

Click on **Main Menu**

Click on **File**.

Click on the filename

Click on **Load**.

Enter **wft**.

The method for acquiring and processing ^{31}P spectra is the same as above except for the setup command (step 6). *See next page.*

Eject your sample, put one of the NMR tubes from the rack in the spinner and insert it properly into the magnet.

When finished with the spectrometer do the following:

Enter **exit** .

Click on **Exit** at the bottom of the screen. Then *Click on* **OK** in the new window.

Useful commands

Acquisition commands

ga - starts acquisition

aa – stops acquisition

wft – performs Fourier transform

Display commands

jexp*x* – join experiment *x* (*x*=1-999 avoid 5). If an error occurs use **cexp(*x*)** command below.

cexp*(#)* – creates experiment area number #

time - display the time an experiment will take

ds - display the spectrum

s1 – save display parameters (can use s1 through s9)

r1 – recall display parameters previously saved with **s1** (can use r1 through r9)

dg – display group (displays parameters)

dg1 – display second (processing, plot... parameters)

dps – display pulse sequence

da – display array

dssh – display stack horizontally

full – uses full screen; needed after **dssh** and other commands

nl – first set cursor close to peak, then finds nearest line

File commands

svf (enter) then supply the filename – to save file with associated data, phasing, etc. or **svf('filename')**

mf(3,4) – copy fid from exp3 to exp4

Processing commands

lb=1 – gives exponential broadening of 1 hertz with **wft** or **ga**

wft – weighted (depending on parameters set) FT

aph – good automatic phase to at least start phasing

cz – zero all integral resets (use menu button **reset** and mouse for resets)

bc – baseline correction with default spline fit; depends on integral resets

Plotting commands

pl – plot spectrum as displayed

pir – plot integral values

pap – plot all parameters

ppa – plot only a few parameters

pscale – plot scale

page – eject page from printer