

NCSU NMR Facility

Using 300 MHz spectrometers

BasicExperiment Set Up

I. Entering the sample into the magnet

- A. Type "ej" to eject the old sample
- B. Remove the old sample and spinner from the magnet
- C. Remove the old sample from the spinner and place the new sample into the spinner
- D. Check the sample with the requirements on the side of the magnet
- E. Place the new sample, which is in the spinner, back on the magnet and hit "enter" on the keyboard

II. Setting the shim and lock

- A. Go to "SET UP"
 - a. Select "frequency" with the left mouse button, and with the right mouse button select and hold the "solvent" submenu
 - b. Release the right mouse button to select the solvent, or type the chemical shift of the solvent (a number)
- B. Hold the right mouse button down on the "set up" button, and then select "shim/lock"
- C. Check that the file temp.shim is being used
 - a. If the file temp.shim is not being used, use the left mouse button to select the current file
 - b. Then type the "filename temp.shim"
 - c. Finally, use the left mouse button and select "get," and it will get the file temp.shim
- D. Display, turn to lock sweep, then adjust the offset so that the lock signal appears in the Center of the screen, then turn the display to off
- E. Turn mode from off to fast
- F. Check that the spinner is on
- G. z2 is operated with the left mouse button, and then z1 is operated with the right mouse button
- H. Adjust z1 until the lock meter goes as high as possible
- I. Then select z2 with the right button and adjust it until the lock goes as high as possible
- J. Finally, select z1 with the right mouse button and adjust it until the lock meter is as high as possible
- K. Repeat steps G-J with z3 with z1, and z2 with z1
- L. If the reading ever is off of the lock meter, adjust the gain so that the lock meter is between 60 and 80

III. Getting the proton spectra

- A. Type "stdh" to get proton spectra
- B. It will ask you how many acquisitions you want, then type the number of acquisitions needed in multiples of 4
- C. When acquiring data has finished, type "save filename", where filename is equal to the name you want to give the file in the defined directory (/home/general/data/[PI]/[user])

Example: /home/general/data/gorman/jennifer

- D. Type "get filename" to get entire spectra.
- E. Type "dis" to display FID
- F. Type "See" to get the Fourier Transform. You will see the spectrum on the screen.
- G. Type "srm" when you are done

IV. Getting the carbon spectra

- A. Type "Stdc1" to get the carbon spectra
- B. Decide how many acquisitions are wanted per pass, i.e. 100 acquisitions per 4 passes, equals 400 acquisitions
- C. It will ask you how many acquisitions you want, then type the number of acquisitions needed in one pass
- D. Then it will ask you how many passes you want, type the number of passes you want to use
- E. Finally it will ask you for a filename to save the data to, type the filename you want it saved under
- F. When acquiring data has finished only the spectra from the last pass is on the screen
- G. Type "get filename" to get the entire spectra, filename is equal to the name you typed in line E of Getting the Carbon spectra
- H. Type "See" to get the Fourier Transform

V. Analyzing the spectra

- A. Hold the right mouse button on the display panel (area containing the spectra) and select "full," to get the entire spectra on the screen
- B. Zooming in on a section of the screen
 - a. Use the left mouse button to select the left end of the screen that is needed
 - b. Use the middle mouse button to select the right end of the screen that is needed
 - c. Hold the right mouse button in the display window and select "zoom"
- C. Purple lines represent expanded spectra, green lines represent full spectra
- D. Type "aph" for autophasing
- E. To manually phase
 - a. Hold the right mouse button down on the "process" button, and then select "phase"
 - b. Use the left button to define the left most peak
 - c. Use middle button to define the right most peak
 - d. Press the left mouse button on the "left" button
 - e. Adjust the A by moving the mouse up and down.
 - f. The phasing is done when the two horizontal lines at the base of the peak are at the same height
 - g. Finally click the right mouse button to turn off the phasing
 - h. Repeat steps d-g, except select the "right" button to phase the right side of the spectra
- F. Finding the integral
 - a. Hold the right mouse button down on the "process" button, and then select "integration"
 - b. Press all three clear buttons with the left mouse button
 - c. Adjust the slope
 - 1. select "adjust slope" with the left mouse button
 - 2. move the mouse up and down so that lines between peaks are as close to horizontal as possible
 - 3. hit the right mouse button to stop adjusting the slope
 - d. Adjust the curvature
 - 1. select "adjust curvature" with the left mouse button
 - 2. move the mouse up and down so that the lines on the peaks are as close to vertical

- as possible
- 3. hit the right mouse button to stop adjusting the slope
- e. Hit "select break points manual" with the left mouse button
- f. Set break points by clicking the left mouse button on each side of the peaks
- g. Press the right mouse button on the display panel to *quit setting break points*
- h. To select integral value
 - 1. place cursor on peak
 - 2. hold the right mouse button down, and then select "set integral value"
 - 3. type the desired value
 - 4. to print select "print"
 - 5. to exit select "OK"

VI. Peak picking

- A. Hold the "process" button with the right mouse button and select "peak picking"
- B. Press the right mouse button on "threshold," and adjust height so that it reaches the minimum height of the peaks that you want
- C. Press the right mouse button to set
- D. Press "find peaks" with the left mouse button to find all of the peaks that are above the threshold

- E. Press "annotate" with the left mouse button to label all of the peaks with their heights

VII. Printing

- A. Select the "hard copy" button, and then select "plot panel"
- B. Choose LW for the laser writer printer
- C. Check data, axis, integral, and annotate if need (there is an error in the program which will not let you deselect annotate unless you exit the hard copy screen)
- D. Check that the LW has a rotation of 0 (or the Zeta has a rotation of 90, if using Zeta printer)
- E. To set the y-scaling, place cursor on largest peak, hold right mouse button and select "set y scaling"
- F. Select "plot" with the left mouse button

Commands for chge300 SUN for NMR

Command	Function
aph	autophase
acq	acquisition
apod_panel	brings up apodization panel
al	automatic lock
bacq	background mode -f -s (#scans) -d (# dummy scans) -p
bcorr	baseline correct
bcorr_panel	brings up baseline correction panel
bc;zerodata -c	zeros data and baseline corrects simultaneously
bigwin	increase size of keyboard win
c (clear)	clears screen
cd	change directory
cp	copy file
cb	change block size
da	gives data acquisition parameters: da -a, da pr -> prints da
deltav	set to one half sweep width for 1-1 Hard Pulse expt.
df	disk free
dg	sets decoupler
digpar	set acquisition parameters -a -b (block size)
dig read	read data to top of stack
dis	displays FID to screen
ej	eject sample (-f =keeps VT and spin rate on)
exp	gets experimental -c, -d
export	file 1 [file 2] -b (for FELIX)
f	full screen
f1, f2	sets frequency(s) +/- x to increase/decrease
ft	Fourier transforms data
ez_panel	brings up easy data acquisition panel
get	gets a data file (-w = in writeable mode)
gn	gain
gamma	gammaH2 decoupling field strength
getshim	gets shim file
getcshim	gets compushim file
lf	gives linewidth of peak
ls	lists files in directory
mv	moves or renames file
na	number of acquisitions
peak_panel	brings up peak panel
peaks	finds peaks

pe	brings up phase panel
pd	pulse delay
plotparm	displays plot parameters
phase	phases data
process_panel	brings up process panel
presat	presaturates sample
printshim lpr	prints shim
pw	pulse width (90*)
rcp	remote copy
rg	receiver gate
rm	removes (deletes) files
rms	calculates root mean square
reverse	reverses spectra
run	begins acquisition
save	saves data as [filename]
see	Fourier transforms data and displays to screen
setinst	sets/removes instrument from data acquisition controlling mode
sg	automatic gain adjustment
smooth	smoothes data
sn	gives signal to noise ratio
spin	gives spin rate
spline	performs cubic spline on data (baseline correction)
splot	brings up panel to
srm	stack remove
ss	sets spin rate
sw	sweep width
tt	tells time experiment will take
za	gives acquisition parameters; za -a, za pr -> prints za
zerodata	zeros data (-c - between cursors)
zg	zero memory, go -> begins experiment

Shim Order for 2D Shimming

Spinning Shims:

Z1, Z2
Z1, Z3
Z2, Z4
Z3, Z5

Nonspinning Shims:

X, ZX,
Y, ZY
 $-Z^2X$, Z^2Y

ZR², R²
X, X³
Y, Y³
ZXY, XY

Misc. Command Routines:

To save an experiment to a filename while running:

run; digread; save [filename]

To look at data while running (only initially):

set experiment parameters (*ie.* za, da, fl)

save [filename]

get -w [filename]

setinst

can change: gn, temp, na

na = x

run

digread -s (sees) digread -b (set to background)

To copy from chge300 to chsparc2:

rcp [filename] chsparc2:/alpha/omega/hardin

To copy from chsparc2 to chge300:

rcp [filename] chge300:/home/omega/data/user/