GN3ØØ-NB Checkout Procedure

Part A: Signal-to-noise (sensitivity) measurement on the quartet of the proton spectrum of Ø.1% ethylbenzene in CDCl₃ (5mm tube).
Note: Underlined texts indicate user entered commands.

Injecting Sample and Setting Up Standard Parameters

1:  >ME 1
Enters the main menu and ejects the sample

2:  Insert the appropriate sample into the spinner, using the depth gauge, and wiping it clean of fingerprints. Set the spinner and sample afloat on the ejecting air flow.

3:  <return>
FREQUENCY (PPM)=x.xx 7.26 <return>
Lowers new sample into probe and LOCK sets the reference frequency (in ppm)
for the lock solvent (cf. handout UGLØ33, ²H solvents).

4:  M
Selects the first nuclei menu page (¹H to ⁴⁵Sc) and takes a few seconds to reset all

5:  1
F: Current Spectrometer Frequency = 3ØØ.511ØØØØ MHz.
Selects ¹H nucleus default values.

6:  M
Return to the main menu.

7:  2
FREQUENCY(PPM)=7.26
If you mistyped or forgot to enter the correct LOCK lock reference frequency while injecting the <return>
sample, you can do it now.

8:  7
RATE xx AIR FLOW xxxx adjust, then <return>
Use knob A to adjust the spin rate to between SPIN 2Ø-3Ø rps. If static electricity is a problem,
touch the grounding plate beneath the Zeta plotter to discharge. Otherwise, the knob
value may be arbitrarily changed by the static charge.

9:  Refer to the procedure "Tuning the GN3ØØ-NB, 1Ø mm ³¹P.¹⁵N Probe", handout UNB332, and tune the probe decoupler channel (HB probe) to ¹H and the lock channel to ²H, on this 5mm ethylbenzene in CDCl₃ sample tube (the observe channel is not being used for part A). After tuning the lock channel, don't forget to check the cable connections and re-select ¹H from the menu.
Locking and Shimming

1: 3 L # <return>
SETTINGS:
Z1    Z2    Z3
5ØØ   5ØØ   5ØØ
<return> Q
COMMAND: X

2: 4 S <spacebar>
"sweep" mode. S acts as a toggle between

3: G M
GAIN(RX) = xxx 8ØØ <return>
desired value).
or G and adjust with knob A

4: T M
TXRF LEVEL = xxx 125 <return>

5: O
of the screen with the offset.

6: P
 signals that are mirror images of each

7: <spacebar> S L
graph and toggle it to the "locked leaving sweep mode.

8: G
8Ø% or 9Ø%.

9: K 1 2
Z1 with knob A and Z2 with knob B
lock level exceeds 100%, adjust the

10: S 1 3
(or <rtn> and K 1 3)

11: <return> <return>

12: X

13: AH ^T T ^S X
light). This will enter the Adjust
the printer (^T), type out the shim
the AH subprogram (X).

Load the shim library (PROBE #) for a 5mm KNOB tube with CDCl₃ as a solvent (or, try each library, after you've locked, and see which gives the highest lock level reading). Type Q to quit and X to exit.

View and adjust the lock display parameters in
the lock bar graph and the sweep mode. The
spacebar in sweep mode will toggle a
"reverse sweep" that is used to phase the
signal.

Set the lock gain to 8ØØ (the M subcommand allows one to manually type in the

Set the lock transmitter power level to 125.

Use knob A to center the signals in the middle

Use knob A to adjust the phase, resulting in positive other.

Toggle the reverse sweep off, return to the fast" mode. It will always be in "standby" lock bar upon

Use knob A to adjust the gain of the bar graph to

Select knob control of Z1 and Z2 shims. Shim on until a maximum lock level is obtained. If the lock level gain as needed with the keyboard knob C.

Repeat the previous step and include Z3. Knob A will control Z1. Knob B will control Z3.

Leave shims and lock display.

Exit menu mode and go to command mode.

Make certain that the printer is "on-line" (green Homogeneity subprogram (>AH), direct the output to values (^T), direct output to the screen (^S), and exit
Preparing for Acquisition

1: >US
   USER: OLDNAME YOUR NAME <return>

2: >WD 1&
   Select the floppy drive. Remove the lab floppy
   from the drive and put it in the pocket

3: >MT
   "DISK ERROR! #5" means that there is no floppy
   in the floppy drive. Put your floppy

4: >MT
   "DISK ERROR! #1101" means that the floppy
   has not been formatted. Refer to
   handout SNB336, "GN300-NB Floppy
   Initialization Procedure" (a copy is under the
   glass near the monitor).

5: If you stopped to format a floppy at this point, your best bet is to reset standard parameters, dial in the shim
   values from the printout, and re-establish the lock. Essentially, you will be repeating most of the steps up to
   this point, but it should not take long to do this. Probe tuning will not be affected and therefore will not need
   to be checked.

6: MT
   If the response is something like "Room for 1559
   more 16384-word files," then WD is
   each time the menu resets default
   set to the hard drive, not the floppy. This will happen
   parameters. How many files ____________
   of what size _______ can the floppy hold?

7: >P2=x.xx 31U
   Set the pulse width to 31 microseconds. This
   that has been determined according to
   360_ Pulse Widths on GE
   is a 90_ pulse width for this sample and probe
   handout UGI032, "Determination of 90_ and
   Spectrometers." This handout may be of use
   to you with your own samples.
This gain value was determined using SG and a delay time of 30 seconds to allow sample, one second would be long

Set the number of acquisitions to one.

Set the delay for one minute to ensure complete relaxation of the sample before pulsing maximum signal-to-noise ratio.

Go acquire data and save the FID under the filename SENS.ØØØ (with an informative comment). Use the down arrow to start the second line of the comment.

Use the arrow keys to adjust the display on the screen.

Set WD up appropriately and copy the data SA, GA = from the floppy to the hard drive (it is faster SB, GB to work up data on the hard drive). Remove BA, BG your floppy from the floppy drive and return GS = x it to it's envelope and box. It is safer there DI, DL = than in the drive. If an old data set by the same name exists, replace it.

Get data from drive 1, which is the floppy drive.
3: >SA
DATASET = xxxxxx.xxx
SENS.ØØØ <return>
COMMENT:
OLD COMMENT
S/N DETERM.Ø.1% ETB <return>
FILE EXISTS! REPLACE? Y

Save data to drive Ø, which is the hard drive.
By saving the data on the hard drive, and
then calling it up from the hard drive, the data
may be processed and plotted more quickly
than from the floppy drive.

4: >GA = SENS.ØØØ <return>

Get the data from the hard drive. Depending
upon how your configured WD, you might use GB
instead.

5: >US = YOUR NAME <return>

Does "USER" have the correct name in it?
(If you've moved to the data station, it may not.)

6: >LB = xxx 1 <return>

Set the line broadening to 1. (The typical
linewidth (in Hz) of a nucleus signal is an
appropriate value to use for line broadening (>LB).

7: >BC

Baseline correct the real and the imaginary FIDs.

8: >EM

Exponentially multiply by the line broadening
value. (Notice the change in appearance of the ends.)

9: >FT

Fourier Transform the FID to a spectrum.

10: >AP

Auto phasing will usually do a close approximation of the needed phase.

11: + or [ [ +

Use the arrow keys to adjust the display on the screen

12: >^N

Toggle to averaged display

13: >PE (adjust phase) <return>
spectrum's phase. (Using knob A, move
knob A, move the second cursor to a the new phase values).

Use the cursors and knobs A and B to adjust the a cursor to a peak on the left end of the spectrum. Using knob B, phase this peak. Using peak on the right end of the spectrum. Phase this peak with knob B. Press <return> to apply
Measuring the Sensitivity.

1: + or [ 
[   + Use the keypad keys to adjust the vertical scale until the quartet is on the screen

2: >MH 
below the top of the quartet Use knob A to set the minimum height cursor

3: <return> Y Y Accept the minimum height entry

4: >YS 
YF = xxxx Defines top and bottom most data points

5: >PP S 
6: O=OP <return> 
Using the Offset command, reference TMS to be zero ppm (if no TMS is present, reference to the center peak of the triplet at 1.242 ppm)

7: >PP S 
8: P 
YF = xxx <return> 
YF represents the peak's height. Measure this peak's height with P, using the bottom point from the YS done earlier.

9: >PM 
already). HZ would go back to Hertz. Change the scale from Hertz to ppm (if it isn't

10: >ZO F 3.5P ^E N ^F <return> 
the region of noise between 3.5 and 5 ppm. The N is measuring the height of the noise.

11: ^T 

12: >SN=xx.x 
number down (i.e., ~25:1). Calculate the signal-to-noise ratio and write the

SN = ____________________
13:  >AM
USE DEFAULT PARAMETERS?  Y
S/N = xx.x
shimming again and re-acquire the
the SN region has a "spike" or

AM = __________.  If AM is less than 3Ø:1, try
data. (AM searches for the smallest region of
noise and compares the signal to it.
Therefore, AM is always a bit larger than S/N. If
"glitch" in it, it will give an erroneously low value).

14:  ^S

Turn the printer off (Screen only).

Plotting the Spectrum.

1:  + or [ Adjust the vertical scale to
[   bring the phenyl region onto the screen

2:  >YS
YF = xxxx
Set the Y-scale for plotting. Typing >YS
informs the plotter of the top and bottom data
points so that it may correctly scale the
spectrum on the paper.

3:  >MH
below the top of the triplet.
<return> Y Y
Use knob A to set the minimum height cursor
Accept the minimum height entry

4:  >PP
L or R
YF = xxxx <return>
Use the L and R keys to position the cursor
on the highest peaks of the triplet and type P P
to get a YF value. Why would we do this?
(This will inform the plotter to make the triplet to
be full scale on the plot.)

5:  >ZO=F1ØP .Ø.5P ^E <return>
Zoom to the full spectrum for plotting

6:  >XY=x 3 <return>
Set the plotting direction

7:  Check that the pen is in the correct position for plotting (about 1cm above the perforated edge of the paper). Press "CLEAR" on the plotter control panel.

8:  >PL
Plot the spectrum

9:  >ZO=F5P 3.5P <return>
Zoom into the noise region. Notice that ^E is
not needed because expand mode has already
been entered.

10:  >RP
PROGRESS!
registered plot.
Do a registered plot of the noise region, Y OFFSET (MM) = x 10 <return> moved up 10 mm and expanded vertically five Y-EXPANSION = x 5 <return> five times

Toggle to the full display (Exit expand mode)

Put the axis, label, and parameters onto the plot.

Wait until LC has finished, then move the pen to an open region on the plot.

Enter the Adjust Homogeneity subprogram
COMMAND: ^T? Direct the output to the printer.
COMMAND: T (shows shims on screen and prints them on the printer).
COMMAND: ^S? Direct output to the screen only.
COMMAND: X Exit the AH subprogram

Plot shim values onto the plot CHARACTER SIZE (1-2) = 1 <return>

Enter the plot text routine
Enter Text: S/N=xx.x,AM=xx.x ^D ^P Enter S/N and AM numbers that you wrote down earlier

Move the paper to the next page

Integration of a $^1$H spectrum of 0.1% ethylbenzene + 1% TMS in CDCl$_3$
Refer to the NMR Basics handout #UGI017 for a discussion of integration.

Assuming one has just finished the sensitivity measurement, the sample has already been locked and shimmed on.

From the menu, reset $^1$H default parameters

Leave the menu and enter command mode

Select the floppy drive

Set number of acquisitions to 32

What is the default P2 ? ______

What is the default D5 value?_____

Scale the gain and record the value ; GN =

Go save one FID and name it ETBINT.000 <return> (with an informative comment)
COMMENT: OLD COMMENT INTEGRATION OF 0.1% ETHYL BENZENE <return> AUTO REPLACE? Y
What is the total acquisition time? ______________

The following may be done on the data station.

1:  >WD  Set up WD appropriately to copy data files to the hard drive and to process them from there (you may wish to use the linked list)

2:  >GA=ETBINT.ØØØ <return>  Call up the data file named ETBINT.ØØØ.

3:  >LB = xx Ø.1 <return>  Enter the line broadening.

4:  >US = NAME?  Is the USER correctly identified?

5:  >BC  Baseline correct the FID,

6:  >EM  exponentially multiply the FID by the line broadening,

7:  >FT  Fourier transform the FID into a spectrum, and

8:  >PS  Phase the spectrum

9:  >^N  Toggle to the averaged display

10:  + or [  Adjust the vertical scale with [ + the keypad keys
11:  >MH  Using the knob, adjust the minimum height to include
the lowest peaks (quartet)

12:  <return> Y Y  Accept the minimum height values

13:  >PP S  Using the L and R keys to
O=ØP <return> position the cursor on the maximum height of TMS, set  TMS to be at Øppm
OR If there is no TMS ...

14:  >PP S  Use L and R to position the
O=1.24P <return> cursor on the maximum
height of the triplet and set it
to be 1.24ppm

15:  >ZO F1ØP-Ø.5P ^E <return> Use zoom to set the region to
be plotted

16:  >YS  Set the Y-scale to define the
top-most and bottom-most
data points for the plotter.
YF could be changed directly to obtain a different plot.

17:  >XY=Ø <return> Set plot size and direction

18:  >PL  Plot the spectrum before
doing any integration

19:  >IS  Display the integral and
scale it (I and D may be used
to increase or decrease the integral size on the spectrum)

20:  + or [ You are in "correction" mode. The up/down arrow keys assigns the keyboard knob C
to the curvature. (Or use knob A). Adjust the "flat" parts of the integral.
The left/right arrow keys assigns the keyboard knob C to the slope correction. (Or use knob B). Adjust the slope to zero.

C

Toggle from knob correction mode to cursor mode

^R

Remove all currently existing "zeroes" with <control> R

F XXP

Position the cursor for a new "zero" at xx ppm

Z

Enter a "zero", or break, in the integration

Repeat the previous two steps until all the desired "zeros" have been entered. A "zero" to the left and right of each peak to be integrated is suggested. (i.e., 8P, 7P, 3P, 2.5P, 1.35P, 1P)

K 3ØØ <return>

After entering a "zero", enter 3ØØ to indicate the relative number of protons (three)

C

Toggle from cursor mode back to knob correction mode.

P

Plot the integral ten

Y OFFSET (MM) = 1Ø <return>

HEIGHT = 17 <return>

<return> make it 17 centimeters high, and then <return> out of the integral display.

>LC

This one command, >LC, will do the following four things: draw an axis (>AX), label the plot (>LA), draw the logo (>LO), and plot the data's parameters (>ZL)

>NP

Move the plot to the next page

Homonuclear Decoupling:

Assuming that the sample is still locked and shimmed from the integration spectrum and that the spectrum is still on the screen, one is ready for homonuclear decoupling.

>GA = ETBINT.ØØØ <return>

If the spectrum isn't on the screen, call it up and process it.

>LB = 0.1 <return>

Process the FID into a spectrum.

>BC

>EM

>FT

>PS

>DC

This DC decoupler power level, DECOUPLER OUTPUT OFF OK? N modulation mode, and post DECOUPLER OUTPUT (Ø - 82DB):=xx63<rtn> delay are the default values and
HETERO DECOUPLING ON OK? Usually work fine.

1=180° SQUARE WAVE 2=90°/180° SQUARE WAVE
3=STANDARD-16 4=CW

MODULATION MODE = 4
POST DELAY (1...57 µSEC):= 9

8: >DN Turn the decoupler on
9: >EF Use the EF subroutine to enter frequencies at which the decoupler will irradiate
10: <control> R Clear the current CD, decoupler offset, list.

Using the knob, position the cursor in the middle of the quartet to be irradiated.

11: D Record the offset:
OF=

12: Using the knob, move the cursor to the triplet
13: D Record the offset:
OF=

14: <return> Exit the EF subroutine

15: >CD = ± <return> <return> <return> Turn on the CD list. Are the correct offsets in the list? (#3 should equal 0 in order to terminate the list)

16: >NA = 32 4 <return> Change the number of acquisitions from 32 to 4.

17: >TT Record the total time needed for the acquisitions:
Time=

18: >GS=2 <return> Go acquire and save to disk
FILENAME= OLD NAME two FIDs that differ by where HMDEC.001 <return> the decoupler is irradiating
COMMENT: They will be called:
OLD COMMENT HMDEC.001 and HOMONUCLEAR DECOUPLING <return> HMDEC.002
AUTO REPLACE? Y

19: >DF Turn the decoupler off after the acquisitions have finished

Processing and plotting may be done on the data station. But first, while still on the spectrometer, continue and see if the desired results occur in step #23.

20: >WD Is WD set up appropriately? Copy the data to the hard drive and process from there

21: LI=GABCEMFTPSSB <return> Set up a linked list to call up INCREMENT NAMES? Y each FID, process it with the same parameters used by the integration spectrum (line broadening and phasing), and save each spectrum

22: >AU = 2 <return> Do the linked list twice,
FILENAME=HMDEC.001 <return> saving the spectra with the DATA SET B=HMDEC.501 <return> same names as the FIDs
AUTO REPLACE? Y except differing by extension number

23: >ZO F4PØ.5P^E <return> Zoom in on the irradiated and decoupled region of the last transformed spectrum

Is the quartet collapsed to a singlet? If not, repeat the acquisition with a larger DC, decoupler output value (more decoupler power). Change DC by 1 unit increments. If one has finished with the decoupler, it should always be turned off with >DF.

24: >GA = ETBINT.ØØØ <return> Get the previously acquired integration data and process it into a spectrum.

26: >BC
27: >EM
28: >FT
29: >PS
30: >ZO = F2PØP^E <return> Zoom in on the region containing the triplet.
31: >YS This will cause this peak to be full scale on the plot.
32: >ZO = F10P-Ø.5P <return> Zoom to a typical proton spectrum size.
33: >CM Use the CM command to adjust the LENGTH = 34 <return> plot size to make room for three spectra on the same plot.
34: HEIGHT = 25.ØØ <return> INTERP ORDER = 1 <return> DASHED PLOT ? N
35: >PL Plot the spectrum.
36: >LC Add the axis, label, and parameters.

It will be necessary to start again with WD and LI if one has moved to the data station.

37: >GA=HMDEC.5Ø1 <return> Call up the first homonuclear decoupled spectrum.
40: >ZO F 4P-Ø.5P^E <return> Zoom to the decoupled region
41: Wait for the previous PL and LC to finish.
42: >RP Do a registered plot of the quartet-triplet region,
Y OFFSET(MM) = 8Ø <return> showing the triplet collapsed to a singlet
Y EXPANSION = 1 <return>
43: >GA=HMDEC.5Ø2 <return> Call up the second decoupled spectrum
44: >RP Do the second registered plot
Y OFFSET(MM) = 16Ø <return>
Y EXPANSION = 1 <return>
45: >CD = _ Turn the decoupler offset list "off"

46: >NP      Move the plot to the next page

47: >^F      Change the spectrum to full scale

79: The next part will begin with ejecting this sample.