HOMODECOUPLING

This handout contains instructions on how to set up a homodecoupling experiment. It originally comes from the U400 Checkout Handout, so should look familiar. Note that this handout guides you through homodecoupling on one specific sample. Pay close attention to the calibration of dpwr, as that is the parameter that will be most sample dependent.

The basic steps for a homodecoupling experiment, discussed in much detail below, are:

1. Select Standard Parameters and Acquire a Preliminary Spectrum
2. Move Homodecoupling Experiment to exp2
3. Set Homodecoupling Parameters
4. Determine Optimum Decoupling Power (dpwr)
5. Set up the Homodecoupling Experiment
6. Start the Homodecoupling Experiment

^{1}H Homodecoupling, 0.1% Ethylbenzene in $CDCl_3$

Select Standard Parameters and Acquire a Preliminary Spectrum

LC Main Menu	select main menu
LC 2:Setup	select setup menu
LC 1:H1,CDCl3	select standard parameters for H1 nucleus in CDCl3
nt=1 <rtn>	set number of transients
gain? <rtn> (should say *gain = Not Used*)	*gain='n'* enables autogain
ga <rtn>	start acquisition and wft when complete

When acquisition is complete,

f full aph <rtn>	display full spectrum to full screen and autophase
dscale <rtn>	display scale
nl rl(7.26p) <rtn>	reference the solvent to 7.26 ppm
f <rtn>	display full spectrum

Move Homodecoupling Experiment to exp2

delexp(2) <rtn> (optional)	delete experiment 2
cexp(2) <rtn> (if exp2 does not exist, *i.e.*, deleted)	create experiment 2
jexp2 <rtn>	join experiment 2
mf(1,2) <rtn>	move fid with parameters from exp1 to exp2

wft <rtn> (NOTE: It is necessary that you do the wft now and not later.)

Set Homodecoupling Parameters

homo='y' <rtn>	select homonuclear decoupling mode
dm='nny' <rtn>	set decoupler to "on" only during acquisition –
gives	decoupled spectra *without* NOE
gain='y' <rtn>	turn off autogain
gain? <rtn>	show gain value selected by autogain during the previous acquisition above

NOTE: There are two reasons for setting gain='y' now. First, the arrayed experiments that you will be doing below will not accept autogain (*i.e.*, *gain='n'*). Second, this will avoid repeating the auto gain setting at the beginning of each acquisition in the subsequent experiments.

Determine Optimum Decoupling Power (dpwr)

dscale <rtn> display scale

Expand around the triplet, then place the cursor at the center of signal and enter:

nl <rtn> select nearest line
sd <rtn> set decoupler offset frequency (*dof*) for the triplet
nt=4 <rtn> set number of transients
dpwr=28,30,32,34,36 <rtn> set up an array for *dpwr*
da <rtn> display the array and double-check the setting
ga <rtn> start acquisition (will wft and display each spectrum sequentially as it is completed)

When acquisition is complete,

ds(1) <rtn> display first spectrum in the array (*i.e.*, *dpwr=24*)

Expand around the quartet, then enter:

ai <rtn> select absolute intensity mode
vsadj <rtn> adjust vertical scale
vs=vs/2 <rtn> set vertical scale to half the current value
dssh <rtn> stacked display the spectra horizontally
pl('all') pap page <rtn> stacked plot the spectra as displayed with parameters

The optimum decoupling power is the *minimum* **dpwr** that achieves complete decoupling of the triplet, *i.e.*, results in the complete collapsing of the quartet to a singlet. This power is depending on the width of the signal being irradiated, *i.e.*, wider signal requires higher power.

Set up the Homodecoupling Experiment

mf(1,2) <rtn> move fid with parameters from exp1 to exp2
wft <rtn> (NOTE: It is necessary that you do the wft now and not later.)

Set Homodecoupling Parameters

homo='y' <rtn> select homonuclear decoupling mode
dm='nny' <rtn> set decoupler to "on" only during acquisition – gives decoupled spectra *without* NOE
gain='y' <rtn> turn off autogain and set gain value to that selected by autogain during previous acquisition

NOTE: This has to be reset since you moved the fid from exp1 to exp2, above, again.

Set *dof* at 5 ppm as the control

dscale <rtn> display scale
LC (at 5 ppm, or type **cr=5p** <rtn>) place cursor at a frequency where there is no peaks within ±1 ppm of that frequency
sd <rtn> set decoupler offset frequency at the cursor position

Set *dof* for the quartet and put it into array

Expand around the quartet, then place the cursor at the center of the signal and type:

sda <rtn> set decoupler offset frequency at the center of the quartet and put it into array

Set *dof* for the triplet and put it into array

Expand around the triplet, then place the cursor at the center of the signal and type:

nl <rtn>	select nearest line
sda <rtn>	set decoupler offset frequency at the triplet and put it into array
da <rtn>	display the dof array (should show the three decoupler offset frequencies set above at about -425, -1365, and -1930 Hz)

Start the Homodecoupling Experiment

dg <rtn>	display dg parameter group
nt=4 <rtn>	set number of transients
ga <rtn>	start acquisition (will wft and display each spectrum sequentially as it is completed)

Enter text while waiting for acquisition to complete.

When acquisition is complete, save the data.

Phase and Display the Spectra

ds(1) <rtn>	display spectrum 1 (the control spectrum)
f full aph <rtn>	display full spectrum to full screen and autophase

NOTE: Autophasing may not work properly due to the glitch at 5ppm. Manually phase the spectrum if necessary.

Manual Phasing

LC 6:Phase	enter the interactive phasing mode

LC (click on a signal toward the left side of the spectrum about halfway vertically up the screen and adjust the phase by moving the mouse vertically while holding down the left button for coarse adjustment, or the right button for fine adjustment, of the zero-order or frequency-independent phase parameter *rp*)

LC (click on a signal toward the right side of the spectrum and adjust the phase as above to change the first-order or frequency dependent phase parameter *lp*)

LC 1:Box	exit the interactive phasing mode

NOTE: if you can't seem to phase the spectrum manually, reset both zero order and first order phases to zero by typing "**lp=0** <rtn>" and "**rp=0** <rtn>", then phase the spectrum again.

wp=2p <rtn>	set width of plot
sp=1p <rtn>	set start of plot
vsadj <rtn>	adjust vertical scale
vs=vs/4 <rtn>	set vertical scale to 1/4 of the current value
dssa <rtn>	stacked display spectra vertically

Stacked Plot the Spectra

pl(1,3) pscale page <rtn>	stacked plot the spectra as displayed with scale