^{1}H HOMODECOUPLING EXPERIMENT

This is a simple and quick means of determining if two resonances are coupled. The HOMODEC experiment is most effective for relatively simple spectra where the couplings are, at least, somewhat resolved. The experiment consists of irradiating a selected resonance with a low power decoupler, which will eliminate any couplings to that resonance. By comparing the resulting spectrum to that without decoupling, it is easily determined which resonance(s) are coupled to the irradiated peak.

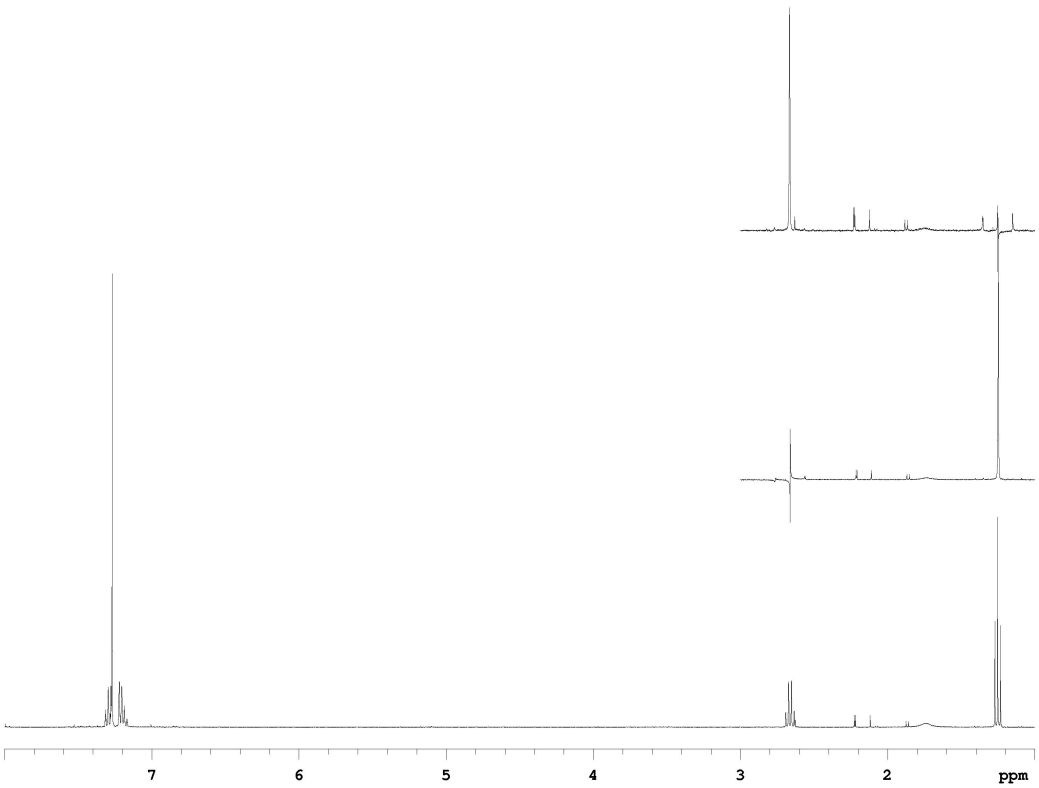

Figure I. An example of homodecoupling on a sample of ethylbenzene in $CDCl_3$. The bottom spectrum is a standard ^{1}H NMR spectrum acquired on a Varian Unity 400 MHz spectrometer. Note the methylene quartet and methyl triplet. The bottom inset (middle, right) shows selective homodecoupling with irradiation at the quartet. The irradiation point typically appears as a 'glitch' in the spectrum. Notice that the methyl triplet has collapsed to a singlet. The top inset shows irradiation at the methyl triplet. Again, notice that the methylene quartet has collapsed to a singlet.

Explanation of Types of Commands Found in this Handout:

1. The VNMR software and the UNIX operating system are both case sensitive. This means that the computer distinguishes whether the letters are entered in upper case (*i.e*. CAPITALS) or lower case. The user must be careful to type the correct case for each letter in a command.
   Example: **jexp1** is not the same as **JEXP1**

2. Some commands are line commands and are typed in by the user followed by a return (a Return⏎ is assumed for typed **bold** text commands).
   Example: **su**

3. Some commands are executed by clicking a mouse button with its pointer on a "button" found on the screen. The execution of these commands are indicated by a two letter designation (LC {left click}, RC {right click}, or CC {center click}) followed by a word or words in shadow or **bold** that would appear in the button.
   Example: LC **Main Menu**

   This means to click the left mouse button with its pointer on the button that says "Main Menu".

4. Some commands are executed by the mouse itself. These commands are indicated by a two letter designation (LC, RC, or CC) and a description of what the user should do in parentheses.
   Example: LC (at 6 ppm)

   This means to click the left mouse button with the mouse cursor positioned at 6ppm.

5. Parameters are entered by typing the parameter name followed by a equal sign, the value, and a return.
   Example: **nt=16**

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Acquire or Load Spectrum to Set Up Standard Parameters

jexp1 join experiment 1.

Acquire and save a ^1H spectrum as normal. Or, if you have one saved,

LC **Main Menu** select main menu.
LC **File Menu** select file storage/retrieval menu.
LC ***desired filename*** select the desired file.
LC **Load** load the selected data to current experiment.

Transform, Display, and Phase Spectrum

wft weighted Fourier transformation.
f full display full spectrum to a full screen.
aph autophase spectrum.

Reference the Spectrum (setting the solvent peak)

LC (at the left side of the solvent region)
RC (at the right side of the solvent region)
LC **Expand** expand region inside cursors.
LC (at the center of the solvent peak)
nl select nearest line.
rl(*your solvent chemical shift*p) reference the solvent peak to the correct chemical shift. The 'p' in the command is required. To reference to $CDCl_3$, type *rl(7.24p)*.

Move Homodecoupling Experiment to exp2

delexp(2) (optional) delete experiment 2.
cexp(2) (if exp2 does not exist, *i.e.*, deleted) create experiment 2.
jexp2 join experiment 2.
mf(1,2) move fid with parameters from exp1 to exp2.
wft (**NOTE**: It is necessary that you do the wft now and not later.)

Set Homodecoupling Parameters

homo='y' select homonuclear decoupling mode.
dm='nny' set decoupler to "on" only during acquisition – gives decoupled spectra *without* NOE.
gain='y' turn off autogain.
gain? 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 autogain setting at the beginning of each acquisition in the subsequent experiments.

Determine Optimum Decoupling Power (dpwr)

dscale	display scale.
ds (if spectrum is not displayed)	enter interactive spectrum display.

Expand *around the a resonance to be irradiated, then place the cursor at the center of signal and enter:*

nl (use only if the splitting is an odd number. Skip if the splitting is even)

sd	set decoupler offset frequency (*dof*) for the resonance.
nt=4	set number of transients.
dpwr=28,30,32,34,36	setup an array for *dpwr*.
da	display the array and double-check the settings.
ga	start acquisition (will wft and display each spectrum sequentially as it is completed).

When acquisition is complete,

ds(1) f full	display first spectrum in the array (*i.e.*, *dpwr=28*).

Expand *around a peak that is coupled to the irradiated signal. Preferably this should be a well-resolved peak. When complete, enter:*

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

The optimum decoupling power is the *minimum* **dpwr** that achieves complete decoupling of the irradiated resonance, *i.e.*, results in the complete collapsing of the coupled partner to a singlet. This power is dependent upon the width of the signal being irradiated, *i.e.*, a wider signal requires higher power. It is the value for *dpwr* you determine in this step that is used below. If complete decoupling is not achieved, you can increase *dpwr* up to a maximum of 45.

Setup the Homodecoupling Experiment

The fid needs to be moved again in order to set the decoupling points. The arrayed data you just collected is not suitable for this purpose.

mf(1,2)	move fid with parameters from exp1 to exp2.
wft	(**NOTE**: It is necessary that you do the wft again.).

Set Homodecoupling Parameters

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

dpwr=*minimum that achieved complete decoupling, above.*

Set *dof* at empty spectral region as the control

ds f full	display full spectrum.
dscale	display scale.

LC (at a region in the spectrum where there are no peaks within ±1 ppm of that frequency)

sd	set decoupler offset frequency at the cursor position.
ds	display interactive spectrum.

Set *dof* for the irradiated resonance and put it into array

f full	display full spectrum.

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

sda	set decoupler offset frequency at the center of the quartet and put it into array.
ds	display interactive spectrum.

Set *dof* for the other resonances to be irradiated and put them into array

f full	display full spectrum.

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

sda	set decoupler offset frequency at the triplet and put it into array.
da	display the dof array.

Repeat for all resonances to be irradiated

Start the Homodecoupling Experiment

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

Enter text while waiting for acquisition to complete:

text('desired text\\more desired text on new line\\etc.')

When acquisition is complete, save the data using either the menus or the svf command.

Phase and Display the Spectra

ds(1)	display spectrum 1 (the control spectrum).
f full	display full spectrum to full screen and autophase.

NOTE: Autophasing may not work properly due to the glitch at the control decoupling point. Manually phase the spectrum if necessary.

Manual Phasing

LC **Phase** enter the interactive phasing mode

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

LC **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** and **rp=0**, then phase the spectrum again.

vsadj	adjust vertical scale.
vs=vs/#	(where **#** is the number of arrayed spectra)
dssa	stacked display spectra vertically.

*If the scale is too large or small, you can adjust it by typing **ds(1**), using the middle mouse to reset the scale, and typing **dssa** to redisplay the stacked plot.*

Stacked Plot the Spectra

pl('all') pscale *pltext* page	stacked plot the spectra as displayed with scale and text in upper left corner of page.

Or to print selected spectra, you;

pl(1,*other desired spectrum #*) pscale pltext page	this will plot the first control spectrum plus the other one you specify. For example, if I want to plot the first and third spectra, I would type ***pl(1,3) pscale pltext page***.

Print Parameter groups

printon *da* dg dg1 dgs printoff (optional)	print parameter groups *with the dof array list*.