Water Suppression using Presaturation (presat)

Presat is a simple two-pulse experiment that utilizes a relatively long, low power RF pulse to selectively saturate a specific frequency, typically water, and a non-selective 45-90º pulse to excite the desired resonances. This pulse sequence is particularly useful for aqueous samples or those with a single large solvent signal. With proper optimization, the resulting spectrum can be mostly free of the solvent signal and lead to improved Signal-to-Noise (S/N) for solute resonances due to the reduction in dynamic range and subsequent increase in available gain. This handout walks you through the optimization of presaturation for water suppression.

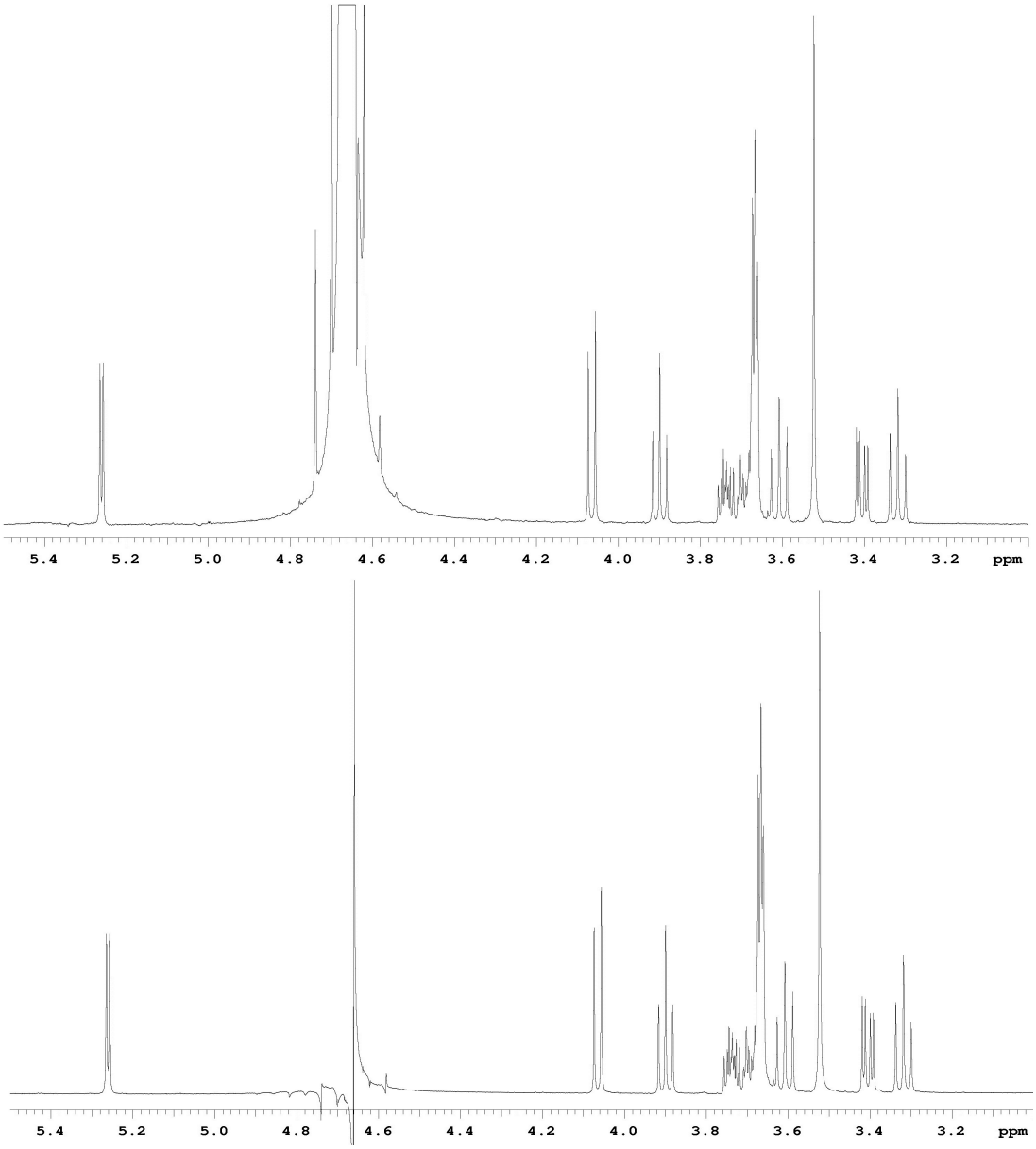

Figure 1. Example of presaturation used on a sample of sucrose dissolved in a D_2O/H_2O mixture. The top spectrum is obtained without presaturation. The bottom spectrum uses presaturation. Both spectra obtained on a UnityPlus-500 spectrometer with 16 scans each.

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 hitting the RETURN key.

   EXAMPLE: **su**

   Hitting the RETURN key is assumed for all bold text.

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 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 the 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 at 6 ppm.

5. Parameters are entered by typing the parameter name followed by an equal sign, the value, and a return.

   EXAMPLE: **nt=16** <rtn>

* * * * * * * *

In exp1, ***setup, lock, shim, and acquire a standard ^{1}H NMR spectrum***. (nt=1 is sufficient).

Still in exp1, ***expand around water peak***, ***place cursor at top***, and type:

nl	move cursor to nearest line
movetof	center spectrometer frequency to cursor
nt=1 ga	submit experiment to acquisition
f full centersw	cursor should be at the top of the water peak. If not, place cursor near top, type **nl movetof ga**. Check if centered on water.
tof?	returns the position of tof
tof =_____	Record the value of tof
mf(1,2) jexp2 wft	move the FID from exp1 to exp2 and join exp2
presat	setup a presaturation experiment
satpwr=5	set the saturation power to 10 dB
satfrq=tof	set the saturation frequency to the transmitter offset
ss=0	set the steady-state scans to zero
gain=20	set gain to 20
ga	submit experiment
av	set display to absolute value. No phase information is displayed
array	setup an arrayed experiment

You will need to answer the following questions, which will appear above the command line:

parameter to be arrayed:	
satfrq	saturation frequency. You want to find the optimum saturation frequency for water suppression
enter number of steps in array:	
20	this is the number of different points you will check for the optimum saturation frequency
enter starting value:	
value of tof determined above – 10	For example, tof=-175.5, then enter –185.5
enter array increment:	
1	this is the step increase between each point
da	displays array. Make sure that the tof is roughly in the center of the array.

ga	start acquisition

Once completed, ***expand around the water peak***

ai dssh	display absolute intensity stacked plots horizontally

Look for the spectrum with the smallest water peak. If the spectra are off scale, type **vs=vs/3 dssh**. Repeat, if necessary. If peaks are too small, type **vs=vs*3 dssh**.

dssl da	lists array numbers and array. Note the array increment that gives the smallest water peak: this is the best satfrq
satfrq=_____(*value determined*)	for example, satfrq=-174.5

Now you will determine the optimal presaturation power. Type,

satpwr=2,4,6,8,10,12	sets the satpwr to an array of values
ga	acquire the arrayed data

Once completed,

dssh	display stacked plots horizontally

Look for the spectrum with the smallest water peak. If the spectra are off scale, type **vs=vs/3 dssh**. Repeat, if necessary.

dssl da	lists array numbers and array. Note the array increment that gives the smallest water peak: this is the best satpwr.
satpwr=*best value from previous step*	set the satpwr to optimal value.
gain='n'	resets gain to automatic control
ph	return to normal phase sensitive mode
ss=4 nt=32 *or higher* ga	acquire a spectrum

Check spectrum for quality of water suppression. Repeat from array of satfrq, if necessary.