H-1 Solvent Suppression: *Frequently used options*

A. Ah1-presat { pre-saturation sequence }. solvent < 50%.
   a. Limitation: all H-1 exchangeable with the Solvent will also be suppressed.

B. Ah1_supress_se { **Pulse Field Gradient Spin Echo sequence**}. solvent > 70%
   a. Limitation: H-1 signals within 0.1ppm region of the solvent will also be suppressed.

C. Ah1-WET Multiple signals (solvents) suppression
   a. Limitation: H-1 signals within 0.1ppm region of the solvents will also be suppressed.

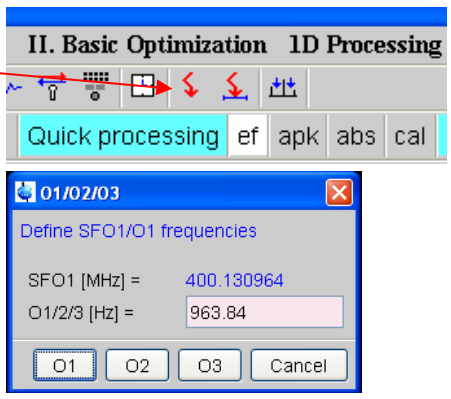
Test sample: a 25mM Sucrose in 70% H2O:D2O solution.

Probe: any high resolution probe with gradient accessory; prefer BBI for higher sensitivity.

Part A: Procedure:

Remark: set and calibrate temperature for dilute samples.

1. Insert your sample.
2. Create a new file, RPAR Ah1 and "GETPRSOL".
3. Tune H-1coil via the "WOBB", with sample non-spinning.
4. Lock, select "H2O+D2O".
5. SHIM: type or select "**TOPSHIM 3D**" instead of TOPSHIM for a complete 3D shim adjustment.
   Comment: 3D shimming takes several more minutes than routine shimming.
6. Set NS = 2
7. Comment: RG is typically 2 or 1, due to large solvent water signal. You will only see the largest signal! See the typical spectrum in page 3.
8. Acquire a routine H1 spectrum. Follow the sequence in the UM set up pull down menu.
9. Click the  in the top menu bar.
10. Place the mouse to the signal of solvent to be suppressed.
    Left click to define the exact location of the transmitter offset.
    Write down the value of offset. O1 (Hz).



Part A (cont'd): Using Solvent pre-saturation pulse sequence.

1) Create a new experiment number "IEXPNO" with the same file name for your sample.
2) RPAR Ah1_presat.
3) GETPROSL and follow the instruction listed in the title page or this handout.
4) Special case: If your sample has high salt or extreme pH, calibrate all the pulses by typing a automation command " pulsecal". Wait till it is completed.
5) Don't type getprosol after "pulsecal".
6) Type O1 and set it to the value obtained from step #8 on page 1. Note "O' is letter o, not zero.
7) Question: do you have to shim or "wobb" again?
8) Set NS to 32, rga, zg to obtain a spectrum.

Part B: Using Pulse Field Gradient Spin Echo sequence.

1) "IEXPNO" to create a new experiment number with the same sample data name.
2) RPAR "Ah1_supress_se"; also named as "Ah1_supress_gp" (same program).
3) GETPROSOL and follow the instruction listed in the title page.
4) Type O1 and set it to the value obtained from step #8 on page 1.
5) Set NS= 32 on the test sample.
6) RGA and ZG to obtain a spectrum.

Part C: Multiple solvents suppression with 13C decoupled

Test sample: 5mg Sucrose octa-acetate in C_6D_6 with ~10 ul of acetone and $CHCl_3$.

Default: spectral width is 16 ppm and offset at 2ppm.

1) Obtain a routine H1 spectrum, use min. NS to evaluate how many large undesirable signals (singlets) to be suppressed.

2) Type "rpar Ah1_WET" and "getprosol".

3) Tune probe (both H1 and C13) and "getprosol".

4) Shim if you have not done it in step 1.

5) To define number of signals for suppression: Type L30 and enter the number of signals from step 1. Default is 2.

6) Define NS (Default is 32).

7) Use automation to start the measurement: type "xaua", but don't walk away!

8) Wait ~ 2-3 min, the automation will halt and prompt to a warning.

9) {"sref": "*Reference peak not found, default calibration done*"}.
Click "close" to accept the warning.

10) The automation will resume and complete the measurement.

Report:

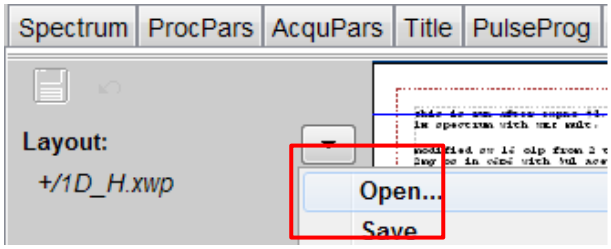
Part A and B: stack plot all three spectra from Part one and two.
Use a layout that will enable you to adjust the scale on each spectrum.

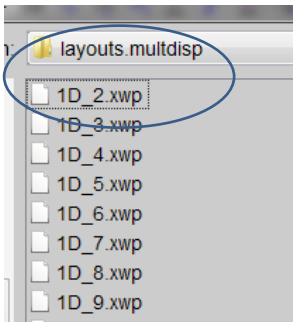
Option of software for stack plots:

A. Old TOPSPIN version : "+/1D+1D+1D.xwp"
B. Newer TOPSPIN 3.2 version "

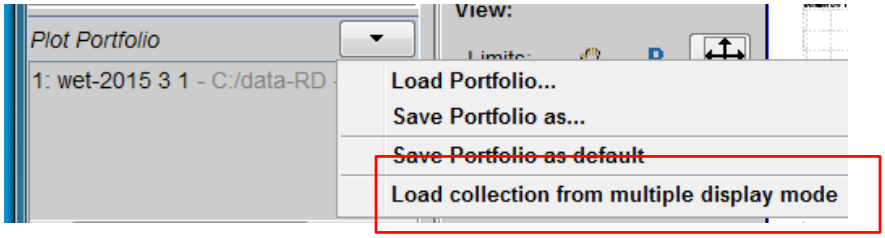
Procedure: Stack plot: using TOPSPIN3.2

1) Display the first spectrum from part two (that uses the pulse gradient method).
2) Click  multiple display icon to define all other two spectra from part one.
3) Exit the multidisplay window, and click plot.
4) Click **Layout**, open and select the **multidisplay** folder, then select "1D_2.xwp" in this example.





5) To import all the spectra from step 1, click Plot Portfolio; then select "Load collection…".



6) All three spectra will be displayed stack, and each spectrum can be modified independently.

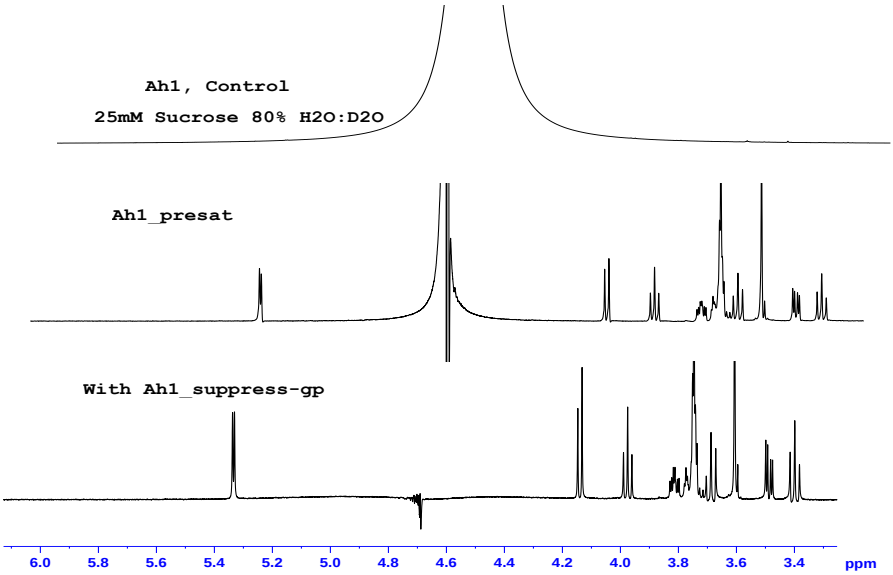
Report: Part C:

Stack plot two spectra using also layouts:

Old version "1D+1D/xwp" or the newer version "+/layouts.multidisp/1D_2xp"

EXAMPLE:

25mM Sucrose in 80% H2O:D2O solution, preferred probe: 400 or 600 MHz H-1 BBI probe



Stack plot from Part C using WET sequence for two signals suppression.

Sucrose octa-acetate in C_6D_6 with acetone and $CHCl_3$.