Homonuclear Decoupling

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Special Note:
This experiment involves setting acquisition parameters that could potentially damage the spectrometer. Do not even think about setting up this experiment without first walking through it with the NMR staff (not someone in your lab who has done the experiment before). **If you damage the probe you will be held responsible for the repair cost and instrument downtime.**

Concept of Homonuclear Decoupling:
Homonuclear decoupling is used to simplify complicated multiplet patterns. Consider the simplest case, a AX spin system. The spectrum of the spin system is a doublet at frequency υ_A and a double at frequency υ_X. In homonuclear decoupling, low power continuous wave irradiation is applied at υ_A. This irradiation has the effect of turning of the scalar coupling for nucleus A. There the spectrum of a homonuclear decoupled AX spin system is a singlet at frequency υ_A and a single at frequency υ_X. In more complicated cases, multiplets can be reduced to more manageable splitting patterns, which simplifies analysis.

How to set up Homonuclear Decoupling in Topspin:
#1) Insert sample into magnet. Lock the spectrometer. Shim. Acquire and process a 1D ^{1}H spectrum.

#2) Copy spectrum and acquisition parameters into a new experiment. In most cases you would have run the 1D ^{1}H in experiment number (experno) 1. To copy the spectrum and parameters into experno 2 type the following at the command line:

wrpa 2

Navigate to this new experiment by typing the following in the command line:

re 2

#3) Setup the new experiment. First we will set up the logical channels of the spectrometer. In the command line type:

edasp

In the middle section under NUC2 select H1 (Fig. 1) and click save.
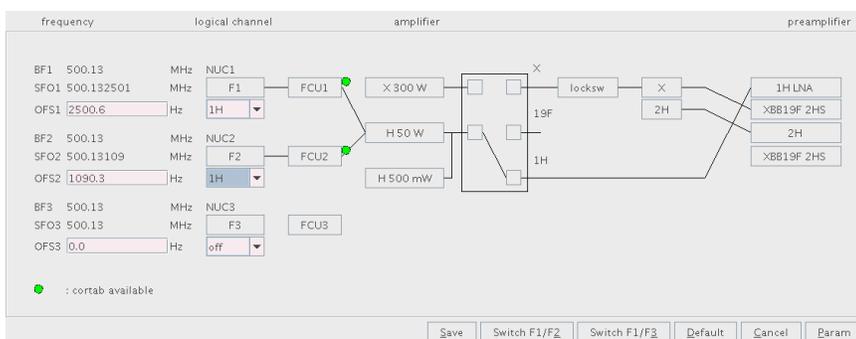

Figure 1. edasp window with logical channel 2 set to proton for homonuclear decoupling experiment.

Next, at the command line type:

pl24

and make sure this power is set to 120 dB.
Next, at the command line type:

DIGMOD

and select homodecoupling-digital (Fig. 2).

Figure 2. Setting the Digitization mode.

Next, at the command line type:

AQ_MOD

and select qsim (Fig. 3)

Figure 3. Setting the Acquisition mode.

Finally, at the command line type:

PULPROG

And select zghd (Fig. 4)

Figure 4. Setting the pulse program.

#4) Zoom in on the peak you want to decouple. Click the button. Center the vertical line on the center of the multiplet and Left click. Save to O2 (Fig. 5)
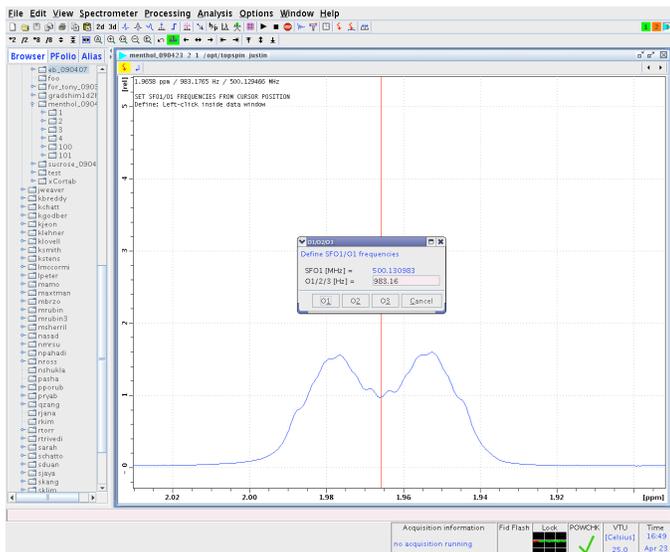
Figure 5. Setting the O2 for homonuclear decoupling. In this case the doublet at ~1.97 ppm is the target for decoupling.

#5) Type zg to run this experiment. This experiment is a control. It is a 1D ^{1}H with homonuclear decoupling at the peak selected in step 4. But there is no decoupling power. Hence this spectrum should look just like the 1D ^{1}H ran in step 1. Note that there will be more noise in this control spectrum (Fig. 6).
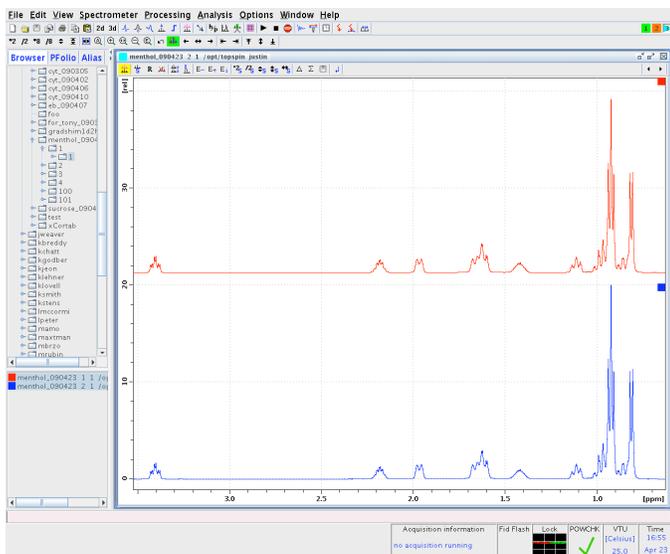

Figure 6. Comparison of the control spectrum for homonuclear decoupling (blue) and the 1D ^{1}H spectrum (red).

#6) Copy the spectrum and parameters into the next experiment (in my case experno 3) by typing the following at the command line:

wrpa 3

Navigate to that experiment by typing the following at the command line:

Re 3

Turn on power to the decoupler. Type the following at the command line:

pl24

and set this power to 70 dB.

It is very important to know that power (in terms of watts) increases as the number of dB decreases. Hence 120 dB is actually 0 watts of power and 70 dB is >0 watts.

Type zg at the command line to run this experiment. Process.

#7) Optimize pl24. The goal of optimizing pl24 is to set the homonuclear decoupling to a value that collapses the multiplet into a singlet. Examine the experiment you ran in step 6. If there is still pseudo-multiplet character, decrease pl24 by 2 dB and run it again. **NOTE WELL: do not decrease pl24 below 40 dB. This could seriously damage the probe!!!** Fig. 7 shows the effects of decreasing pl24 on a doublet.
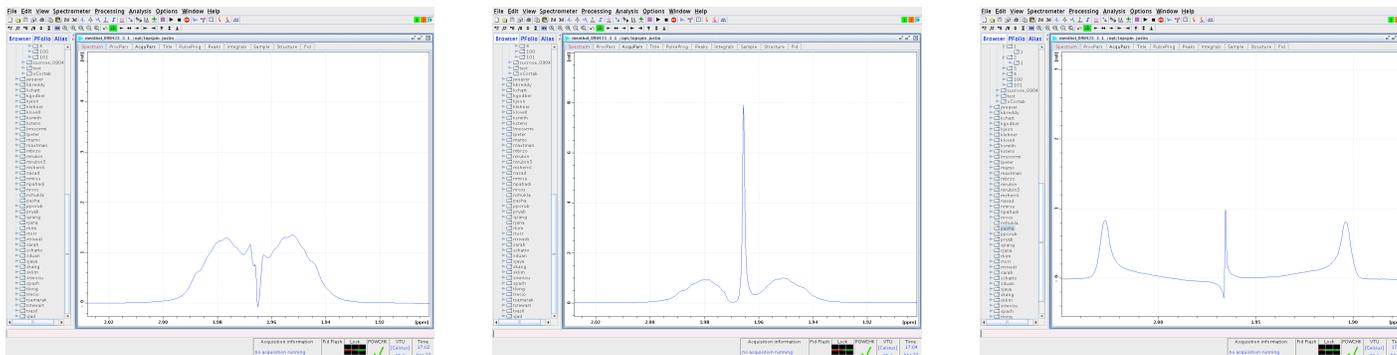

Figure 7. 1D homonuclear decoupled spectra with pl24 set to 70 dB (left), 50 dB (middle) and 40 dB (right). Note that the decoupling does not improve significantly by decreasing pl24 below 50 dB.

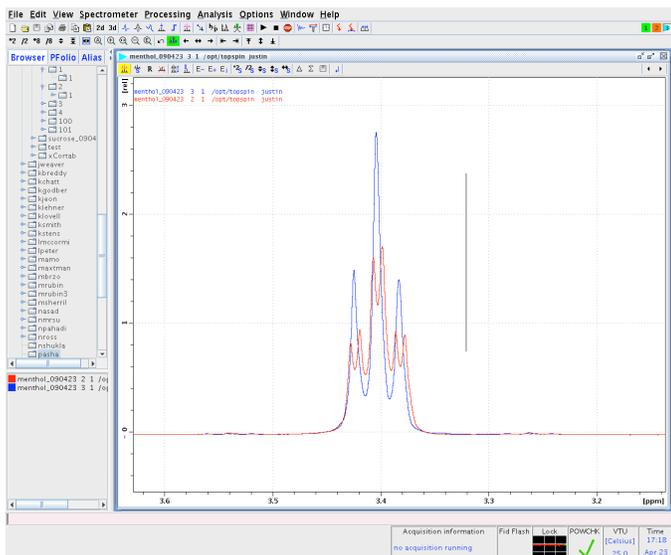A comparison of the control spectrum and the homonuclear decoupled spectrum is shown in Fig. 8.

Figure 8. A comparison of the splitting of a peak at ~3.4 ppm without (red) and with (blue) homonuclear decoupling at ~1.97 ppm. Note that the resonance at ~3.4 ppm is a doublet of triplets with the smaller splitting due to the coupling to the resonance at ~1.97 ppm. Once this resonance is decoupled (Fig. 7) the doublet of triplet is reduced to a triplet, which simplifies analysis.