

The GOESY

Justin Douglas (justindouglas@ku.edu) – KU NMR Labs v1.1 7/14/09

Special Note:

This experiment involves setting acquisition parameters that could potentially damage the spectrometer. Do **not** attempt to set up this experiment without first walking through it with Justin.

Also this experiment is only calibrated on Paris (the walkup 500 MHz spectrometer in Malott B042). Do **not** attempt to set up this experiment on another spectrometer.

Concept of the GOESY:

The GOESY (gradient enhanced nuclear Overhauser effect spectroscopy)¹ is used to assess the spatial proximity of two or more protons in a molecule. This experiment employs excitation sculpting² to selectively invert a target resonance. This selective inversion perturbs the populations of other spins close in space (< ~5 Å) to the target resonance.

How to set up the GOESY in Topspin:

#1) Record a 1D ¹H NMR spectrum of your molecule (Fig 1). Pick your target resonance. Because this experiment uses selective pulses, it is important to choose an isolated peak. Make sure there are no peaks within ± 50 Hz of your target resonance. Determine the frequency (in Hz) of your target resonance. To read the frequency of the target resonance, right click on the spectrum and choose “Display Properties”. Turn on “Cursor Information” (Fig. 2)

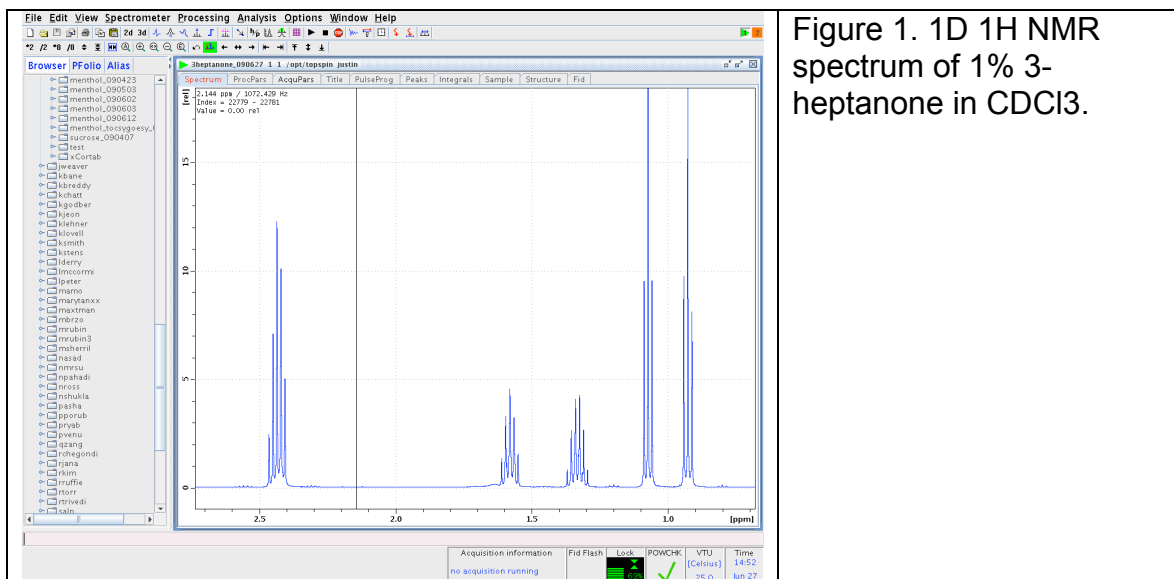


Figure 1. 1D ¹H NMR spectrum of 1% 3-heptanone in CDCl₃.

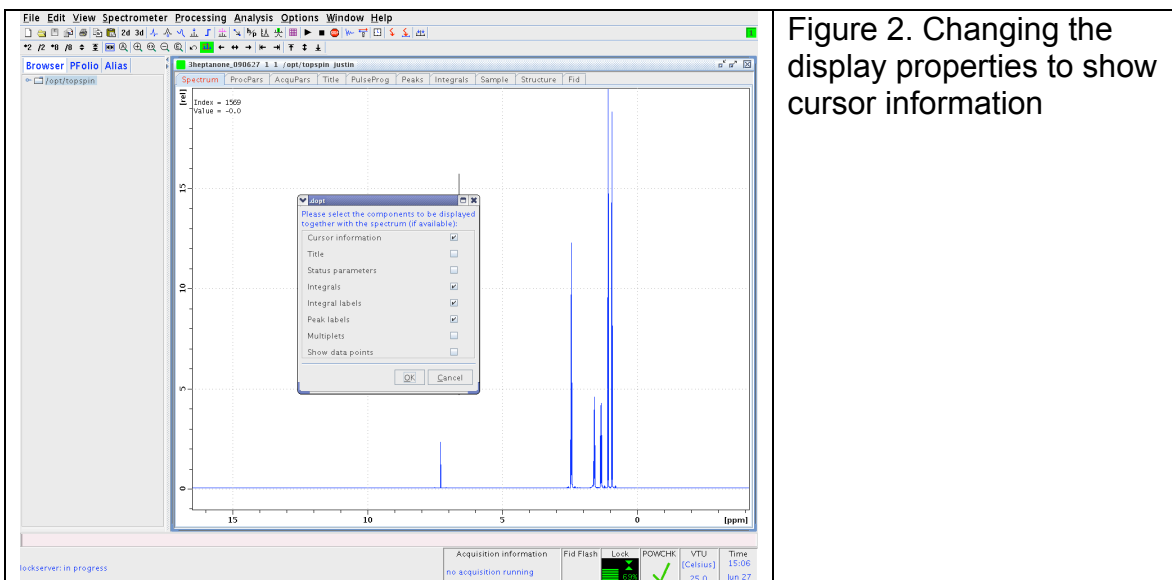


Figure 2. Changing the display properties to show cursor information

In the case of this example, the target resonance is the triplet at ~0.9 ppm, which equals 463.7 Hz.

#2) Create a new experiment by typing “edc” at the command line. Choose SELNOGP as the experiment (Fig. 3).

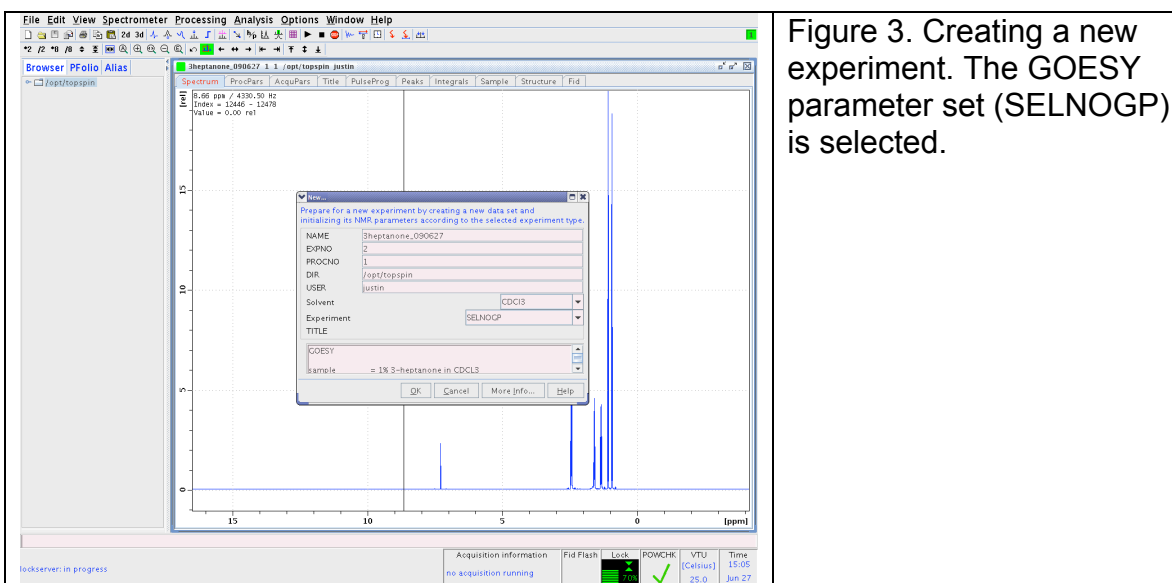


Figure 3. Creating a new experiment. The GOESY parameter set (SELNOGP) is selected.

Type “getprosol” at the command line to read probe-specific parameters. Check the parameters by typing the name at the command line and comparing the value in the popup with the table below.

Table 1. Parameters for the GOESY experiment

| Parameter | value | notes |
|-----------|-------|-------|
|-----------|-------|-------|

| | | |
|--------|-------------|--|
| name | | |
| gpz1 | 15.0 | |
| gpz2 | 40.0 | |
| spnam2 | Reburp.1000 | |
| sp2 | 48.96 | Check the prosol table. Ask Justin for correct values. |
| p12 | 116280 | Check the prosol table. Ask Justin for correct values. |
| ds | 4 | |
| ns | 32 | This value depends on your molecule |
| o1 | 3088.51 | |
| d1 | 5 | |

#3) Set the position of selective pulse. This parameter is called spoffs2 and it equals the position of the peak minus o1. In this example, spoffs2 equals -2624.3 (Figure 4).

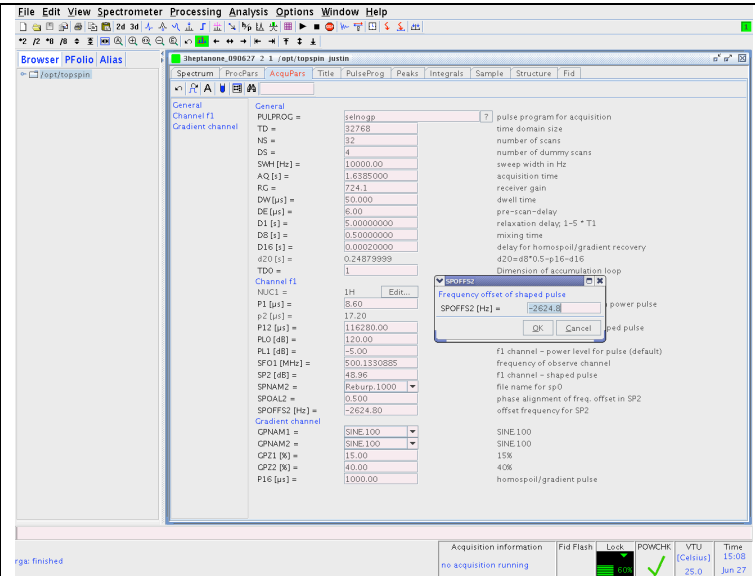


Figure 4. Setting spoffs2. Note that this step is absolutely critical. If this value is not set correctly to the target resonance, then the results will be poor.

#4) The KU NMR lab highly recommends that you run this experiment with multiple mixing times (d8=0.025, 0.05, 0.1, 0.2, 0.5, 1, 2 s). Set the initial d8 by typing "d8 0.025". Use the command "iexpno" to clone the current experiment and set the next mixing time by typing "d8 0.05". Continue this process for every d8. Navigate to your experiment with the largest d8 and type "rga" at the command line to set the receiver gain. Navigate to the first experiment and type "multizg" and enter the number of experiments to start the queue. Once the experiment is done type "ef" to process the data. Phase the data by hand so that the target resonance has negative intensity (Fig. 5). Protons in close proximity to the target resonance have positive intensity. Depending on the

molecule and the mixing time, some peaks will not be in-phase (for instance the peak at ~1.35 ppm in Fig. 5).

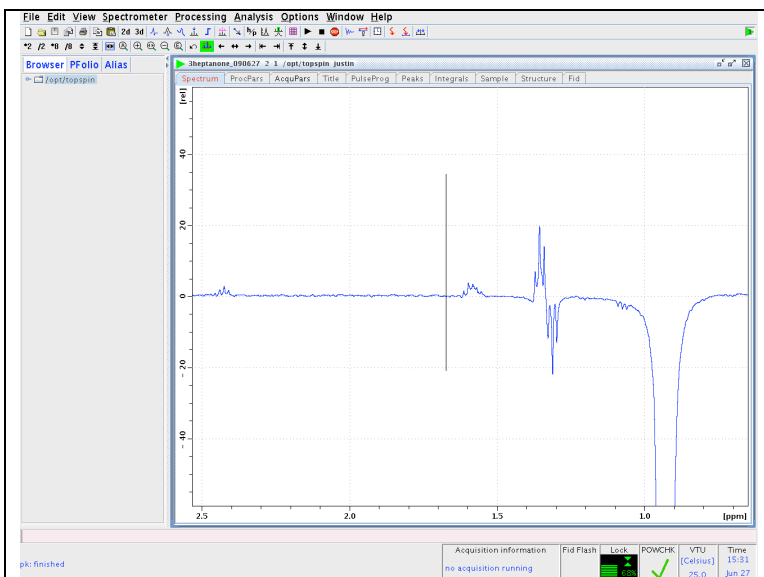


Figure 5. 1D GOESY spectrum of 1% 3-heptanone in CDCl_3 with a mixing time of 500 ms. Notice that the target resonance is phased to be negative. Peaks at ~1.6 and 2.45 show weak NOEs. The peak at ~1.35 ppm is not in-phase, possibly because the mixing time is too long and this peak is scalar coupled to the target resonance.

#5) The intensity of the positive peaks depends strongly on the mixing time, d_8 . Decreasing d_8 will reduce the size of the positive peaks and show only peaks that are very close to the target resonance. Increasing d_8 will increase the size of the positive peaks and show peaks that are further away from the target resonance. It is recommended that you run this GOESY experiment with several mixing times between 50 ms and 2 s to avoid misinterpreting your results (Fig. 6)

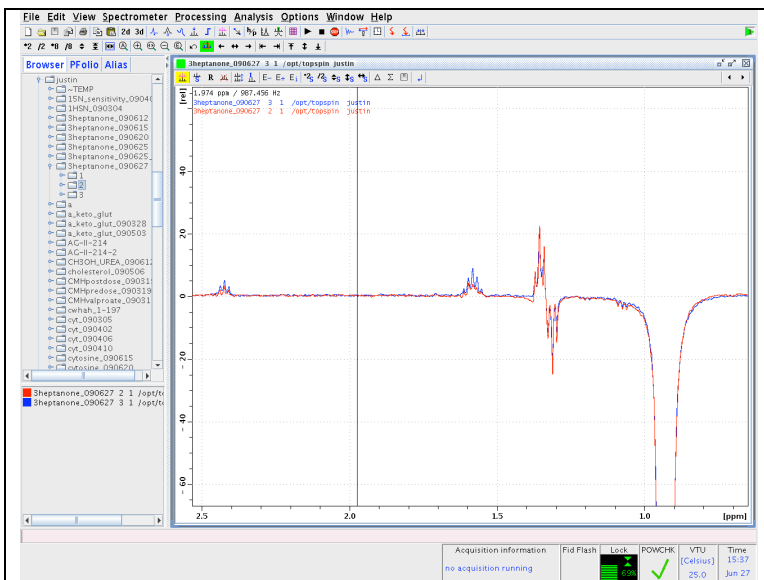


Figure 6. Overlay of 1D GOESY with mixing times of 500 ms (red) and 1 s (blue). Notice the increase in intensity of the peaks at ~1.6 and 2.45 ppm.

References

¹Stonehouse *et al.* JACS (**1994**) 116, 6037.

²Stott *et al.* JACS (**1995**) 117, 4199.