1D TOCSY

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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 in 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 1D TOCSY:

The 1D TOCSY experiment elucidates all protons in a network of coupled spins. Consider three protons in complicated molecule, H^A, H^B and H^C, where H^A and H^C are coupled only to H^B. Likewise, H^B is coupled only H^A and H^C. The 1D TOCSY of this molecule with selective excitation of H^A will only show signals for H^A, H^B and H^C. Hence, this experiment is a powerful tool for simplifying complicated spectra.

How to set up the 1D TOCSY in Topspin:

#1) As a control acquire a 1D ^{1}H NMR spectrum of your molecule (Fig. 1). Examine this spectrum and choose an isolated signal for selective excitation. This signal should not have any other peaks within ± 50 Hz. In the case of 3-heptanone used as an example in this protocol, I will choose the two upfield triplets at 0.93 ppm (464.2 Hz) and 1.07 ppm (536.9 Hz). To read the frequency of the target resonance, right click on the spectrum and choose “Display Properties”. Turn on “Cursor Information” (Fig. 2)

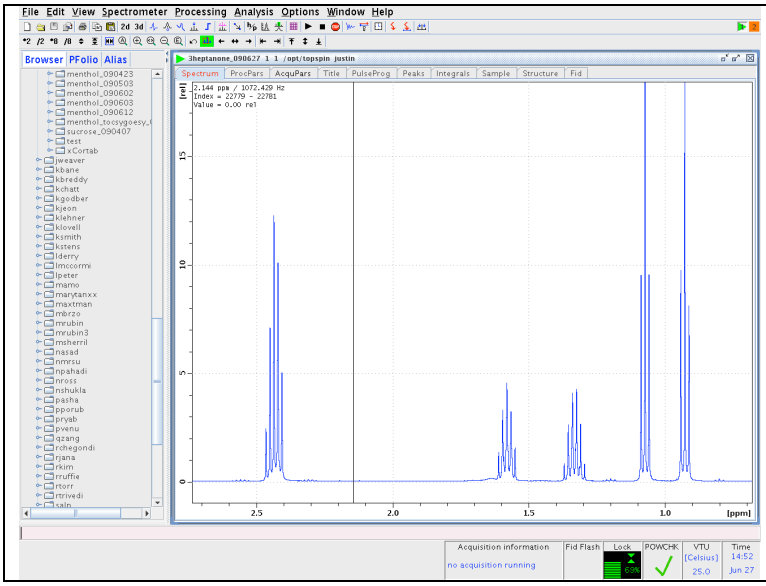

Figure 1. 1D 1H NMR spectrum of 1% 3-heptanone in CDCl3.

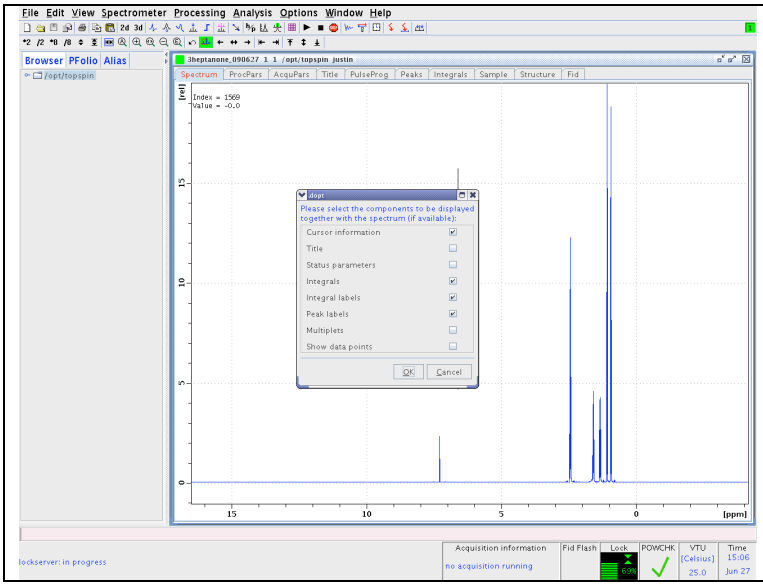

Figure 2. Changing the display properties to show cursor information

#2) Create a new experiment by typing “edc” at the command line. Choose SELMLGP as the experiment. 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 1D TOCSY experiment

Parameter name	value	notes
p6	25 μs	low power 90° pulse width for TOCSY spin-lock.
pl10	4.27 dB	power level for TOCSY spin-lock.
p12	116280 μs	pulse width for selective excitation
sp2	48.96 dB	power level for selective excitation
spnam2	Reburp.1000	shaped pulse for selective excitaion.
o1	3088.51 Hz	
d9	70 ms	TOCSY mixing time

#3) Set the offset for selective excitation. This parameter is called spoffs2 and it equals the position of the target resonance (in Hz) minus o1. In the case of 3-heptanone if I wish to excite the upfield triplet at 0.93 ppm (464.2 Hz) I would set spoffs2 equals -2624.3 = 464.2 Hz – 3088.5 Hz (Fig. 3).

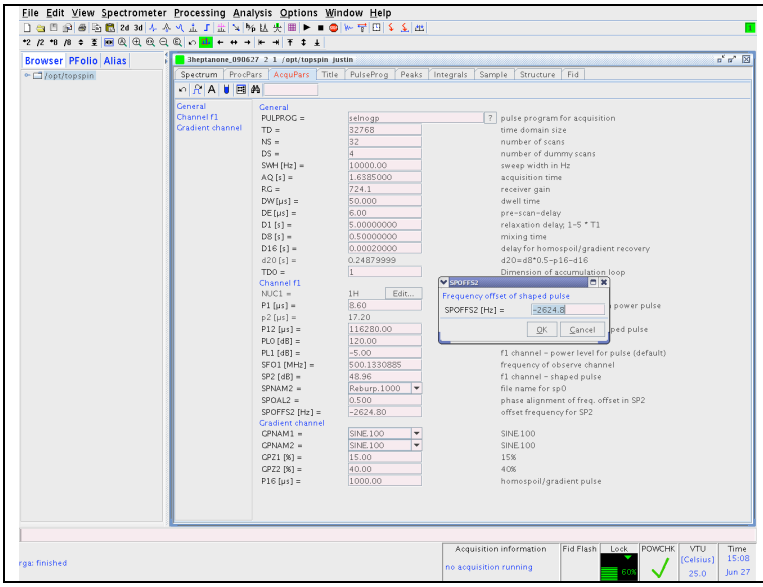

Figure 3. Setting spoffs2. Note that this step is absolutely critical. If this value is not set correctly to the target resonance, then the results will be poor.

#4) Type rga at the command line to set the receiver gain and than zg to start the acquisition. Process the spectrum with ef and apk. Fig. 4

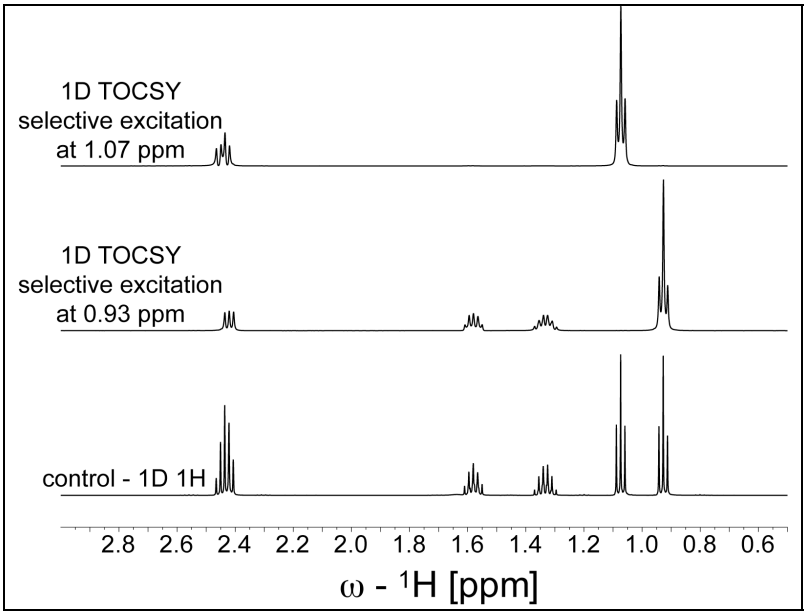

Figure 4. 1D TOCSY spectrum of 1% 3-heptanone in $CDCl_3$ with a mixing time of 70 ms with selective excitation at 1.07 ppm (top), 0.93 ppm (middle) and 1D ^1H control (bottom).

References

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Poppe and van Halbeek J. Magn. Reson. **1992** *96*, 185.

Facke and Berger J. Magn. Reson. Ser. A. **1995** *113*, 257