

## Technical Bulletin #C0040: TOCSY with CapNMR

*This bulletin describes how to use the CapNMR probe for Tocsy ( $^1\text{H}$ - $^1\text{H}$ ) on a Varian console/system.*

Begin by loading an appropriate 'Proton' sequence. Make sure to use the appropriate power levels (see Figure 1). The solenoid microcoil in the CapNMR probe requires less transmitter power than traditional probes. The standard pulse width (pw90) is calibrated at installation for a transmitter power of 45 dB (tpwr=45).

Parameter	Varian Range	Bruker Range
Lock power	1 to 8	-55 to -45
Lock gain	34 to 48	110 to 130
Transmitter power level (dB)	40 to 45	15 to 20
90° Transmitter pulse width (µsec)	2.5 to 5	2.5 to 5
90° Decoupling pulse width (µsec)	5 to 12	7 to 12

Figure 1. Appropriate power levels for the CapNMR Probe

Once there is a good Proton spectrum, type the macro 'Tocsy' into the command prompt and this will load a slightly modified TOCSY pulse sequence. You can view the pulse sequence with a dps command. The sequence should look like the sequence in Figure 2.

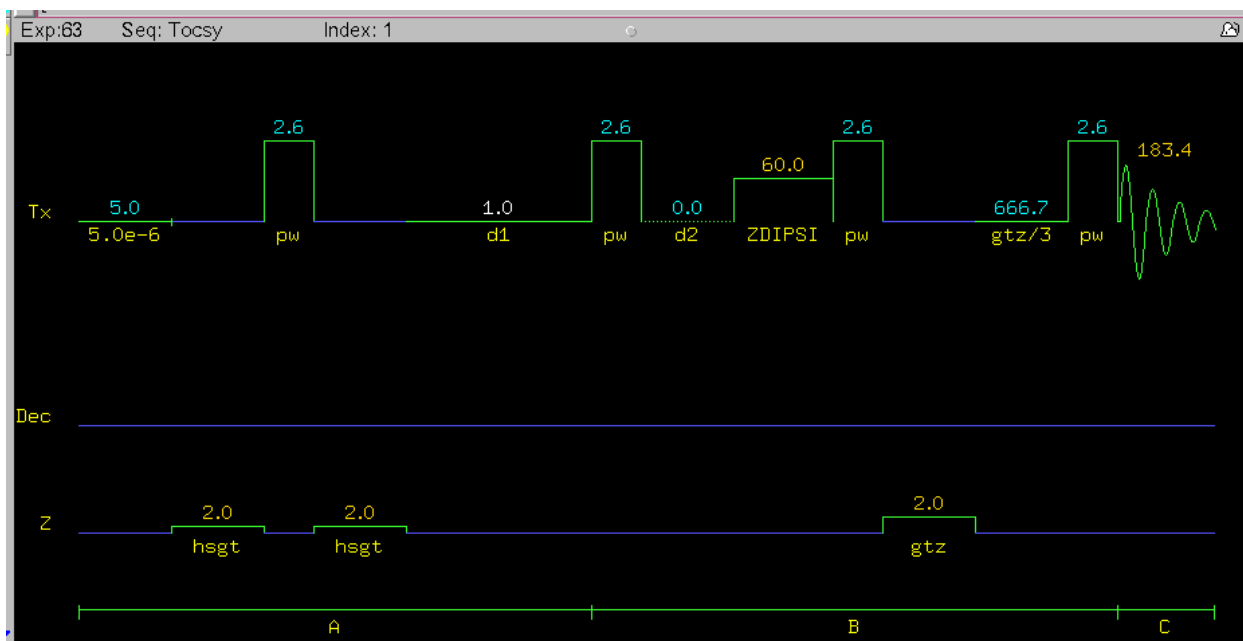


Figure 2. Tocsy Pulse Sequence

However, the pulse width is probe/console specific so your pulse width (pw) value should be different than the 2.6 value shown in Figure 2 and Figure 3. The pw90 for proton was calibrated with an array of the pw parameter in a Proton sequence and for tpwr=45, the pw90 for this probe was 2.6 microseconds.

Here is an example of the parameter set used for the Tocsy pulse sequence. In order to view this window, click on Process and select Text Output and then type 'dg'.

	ACQUISITION	TRANSMITTER	FLACS	PRESATURATION
seqfil	TOCSY	tn	H1 hs	n satmode nnn
sw	5584.6	sfrq	599.763	sspul y satfrq 499.8
at	0.183	tof	-311.8	PFGflg y satdly 0
np	2048	tpwr	45	hsglvl 888 satpwr -13
ss	16	pw	2.650	hsgt 0.002000 PROCESSING
dl	1.000	tpwr_cf	1.000	SAMPLE gf 0.085
nt	20	DECOUPLER	date	Sep 14 2006 gfs not used
ct	20	dn	C13	solvent CDC13 fn 2048
	2D ACQUISITION	dm	nnn	sample 2D PROCESSING
swl	5584.6	SPIN LOCK	SPECIAL	gfl 0.021
ni	128	spinlock	7818	temp not used gfs1 not used
phase	arrayed	mix	0.060	spin not used procl 1p
		trim	0.0020	gain 54 fnl 2048

Figure 3. Tocsy Parameters for the CapNMR Probe

**IMPORTANT: GRADIENT POWER LEVELS**

The CapNMR probe has a much stronger z-gradient than most standard tube probes. Most common tube probes are 50-80 gauss/cm at 10 amps while the CapNMR is commonly 350-400 gauss/cm at 10 amps. The default hsglvl value from Varian is 2000. You may have seen this value as high as 4500 for some tube probes. However, due to a more efficient gradient coil, not as much gradient power (hsglvl) is required for the same gradient field.

**A standard gradient value (hsglvl) for the CapNMR probe is 700 to 900.**

These gradient and homospoil gradient values are automatically calculated from the gcal value in the probe file. The gcal value is calibrated at installation with a profile sequence, which performs a z gradient echo, and is saved in the probe file. Make sure your probe is set correctly (check with 'probe?') and that the 'probegcal' value is correct.

*Sample and Data Set:*

The sample used was 10% (v/v) 2-ethyl-1-indanone in CDCl<sub>3</sub>. Pure 2-ethyl-1-indanone is a clear, brown liquid with a T1 relaxation of approximately 0.8 seconds. This data set was taken on a **5-μL of 10% 2-ethyl-1-indanone (519 μg; 3.2 μmol; v/v in CDCl<sub>3</sub>)**, which was injected and then pushed into a 5 microliter flowcell with more CDCl<sub>3</sub> solvent. This data was taken on a Varian Inova at 600 MHz with a TXI (Triple Inverse <sup>1</sup>H, <sup>2</sup>H, <sup>13</sup>C, <sup>15</sup>N) CapNMR probe with a 5 microliter enhanced flowcell in 1.8 hours.

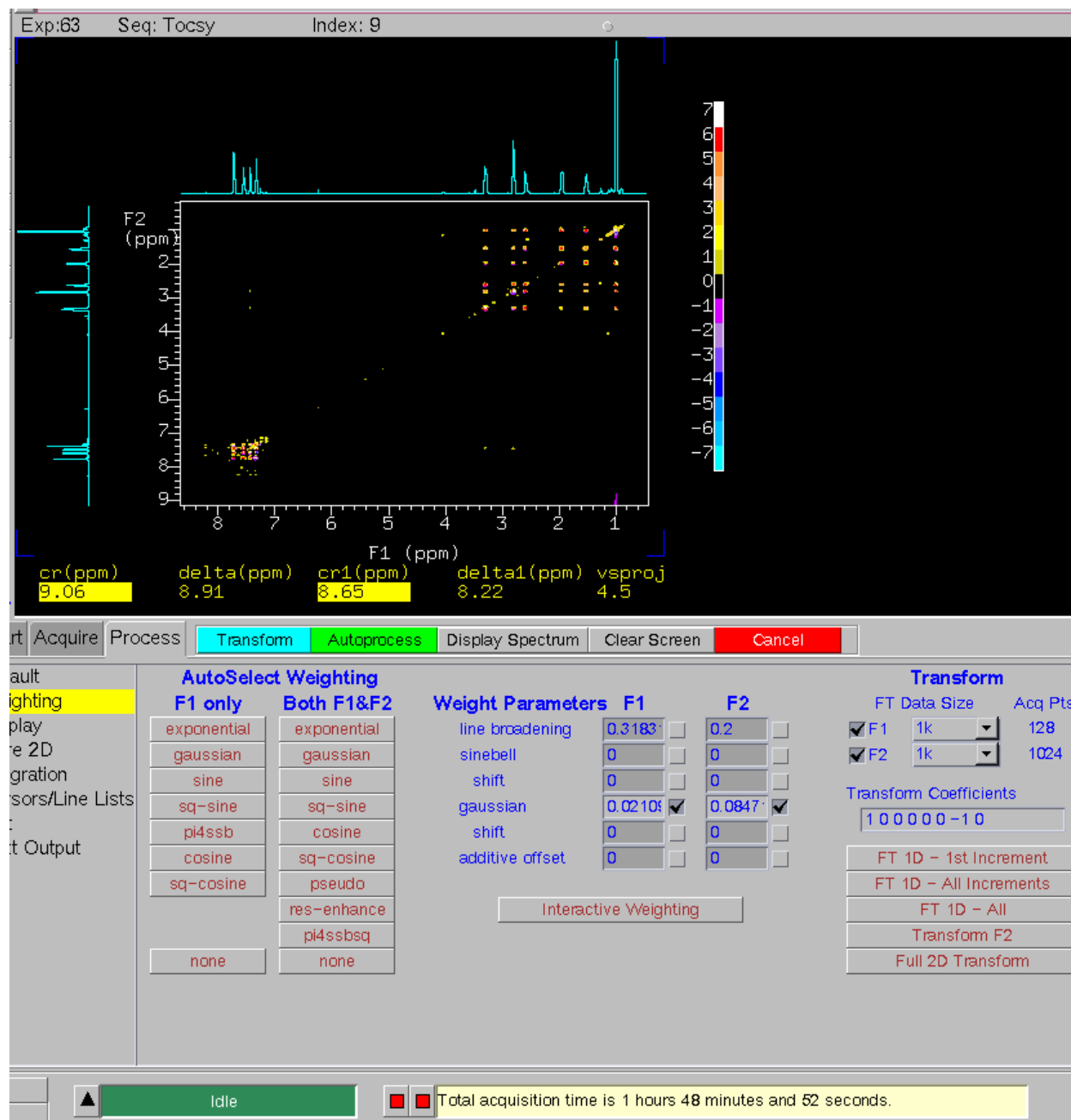


Figure 4. Tocsy Spectrum from 10% 2-ethyl-1-indanone

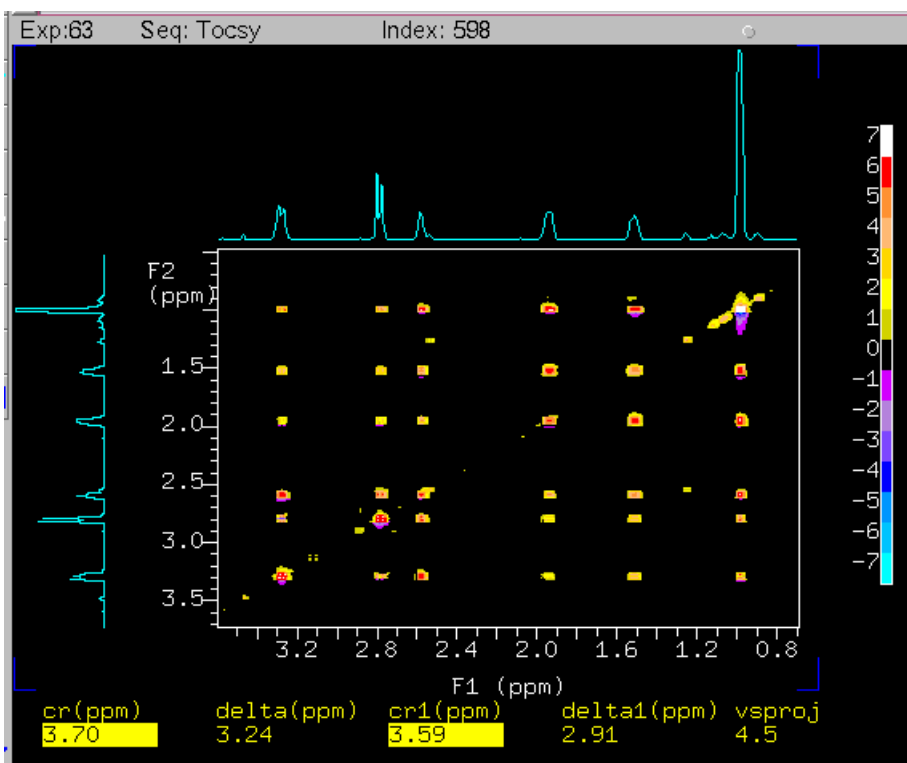


Figure 5. Tocsy Spectrum (Aliphatic Region) from 10% 2-ethyl-1-indanone

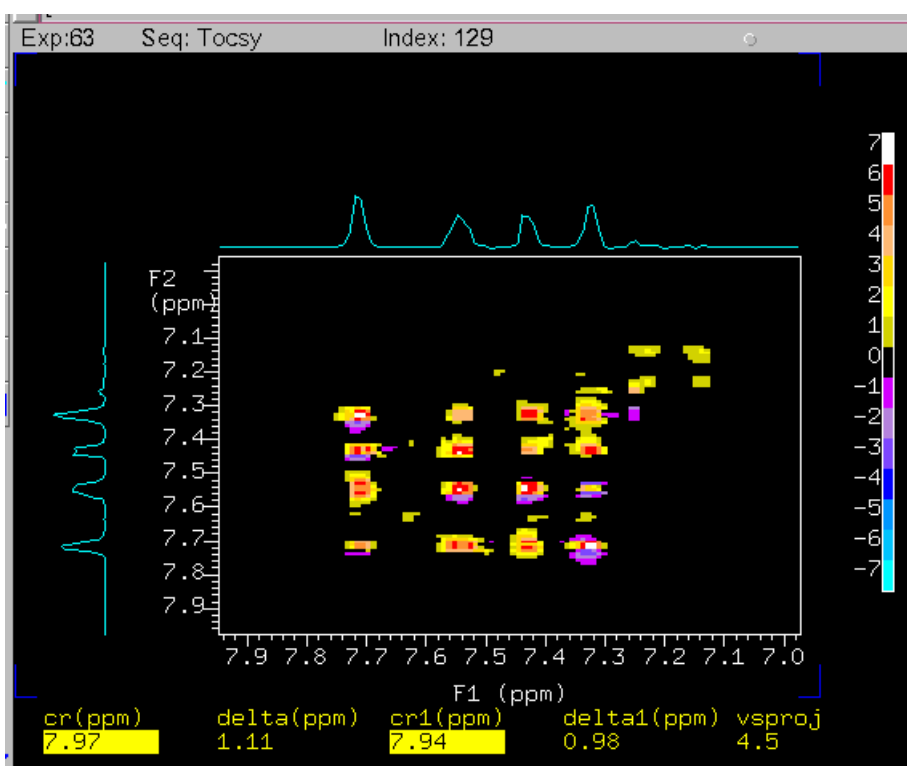


Figure 65. Tocsy Spectrum (Aromatic Region) from 10% 2-ethyl-1-indanone

### *Injection Method*

A Hamilton Gastight 25- $\mu$ L syringe was filled with clean  $\text{CDCl}_3$  solvent and used to rinse the probe. Then, 5- $\mu$ L of 10% 2-ethyl-1-indanone (519  $\mu$ g; 3.2  $\mu$ mol; v/v in  $\text{CDCl}_3$ ) was drawn into the syringe and injected into the probe. Using the same syringe, 12  $\mu$ L of clean  $\text{CDCl}_3$  was picked up and injected into the probe to push the 5- $\mu$ L sample into the NMR flow cell of the CapNMR probe. The 12- $\mu$ L Push Volume was calibrated in advance of sample injection.

### *Screenshots*

All of the screenshots shown in this Technical Bulletin are from a Varian Inova console running Varian VnmrJ 2.1B software on a Unix workstation. The magnet was an Oxford 600 MHz magnet. (For more information on the Varian VnmrJ software, see <http://www.varianinc.com/cgi-bin/nav?products/nmr/>)