Technical Bulletin #C0052: NOESY1D with CapNMR

This bulletin describes how to use the CapNMR probe for Noesy1D 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 'NOESY1D' into the command prompt and this will load a 1D NOESY parameter set. ***First, make sure the pw90 and tpwr settings are appropriate.*** With the Proton spectrum still in the memory, view the spectrum and place your cursor on either side of the peak you want to selectively excite (as close as possible to the peak base). Click on the Acquire tab and click the [Select] button. If you want to excite more then one area, place your cursor around that peak as well and click the [Select] button. Once you've selected the peak(s) you want to excite, click on the [Proceed] button which will calculate the selpw and selshape (using the pw90 and tpwr settings).

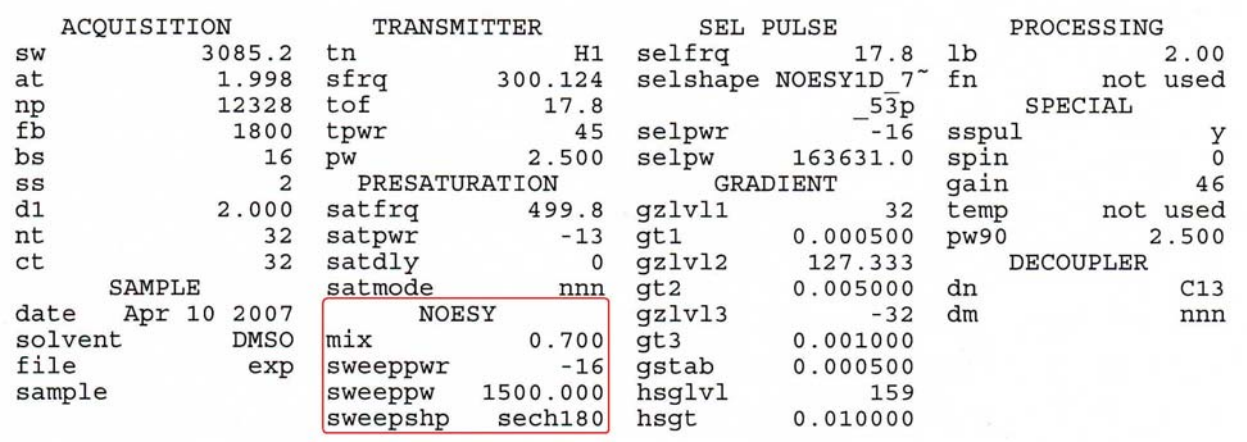
IMPORTANT: Sweep power level

The microcoil inside the CapNMR has a surprising efficiently when it comes to sweeppwr. A normal 5 mm tube has a sweep power of 45 dB but the CapNMR uses the power much more efficiently so it only requires -16 dB for it's sweepwr.

IMPORTANT: Noesy Mixing Time

The mixing time ('mix') is a very important component of the Noesy experiment. The mixing time is split 70/30 in the pulse sequence; for example, a total mixing time of 'mix=1.000' second would be split into 700 millisec. and 300 millisec. **The mixing time is sample specific** and can greatly affect your signal-to-noise.

Here is an example of the parameter set used for the NOESY1D pulse sequence. In order to view this window, click on Process and select Text Output and then type 'dg'.



ACQUISITION		TRANSMITTER		SEL PULSE		PROCESSING	
sw	3085.2	tn	H1	selfrq	17.8	lb	2.00
at	1.998	sfrq	300.124	selshape	NOESY1D_7~	fn	not used
np	12328	tof	17.8		_53p	SPECIAL	
fb	1800	tpwr	45	selpwr	-16	sspul	y
bs	16	pw	2.500	selpw	163631.0	spin	0
ss	2	PRESATURATION		GRADIENT		gain	46
d1	2.000	satfrq	499.8	gzlvl1	32	temp	not used
nt	32	satpwr	-13	gt1	0.000500	pw90	2.500
ct	32	satdly	0	gzlvl2	127.333	DECOUPLER	
SAMPLE		satmode	nnn	gt2	0.005000	dn	C13
date	Apr 10 2007	NOESY		gzlvl3	-32	dm	nnn
solvent	DMSO	mix	0.700	gt3	0.001000		
file	exp	sweeppwr	-16	gstab	0.000500		
sample		sweeppw	1500.000	hsglvl	159		
		sweepshp	sech180	hsgt	0.010000		

Figure 3. NOESY1D Parameters for the CapNMR Probe

IMPORTANT: GRADIENT POWER LEVELS
The CapNMR probe has a much stronger z-gradient then 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.

Example Data Set:

Here is an example NOESY1D spectrum of Quinine in 5 minutes. The negative peak at ~7.5 ppm is the peak that was selectively excited which generally has an amplitude 10x greater than its noe correlating peaks.