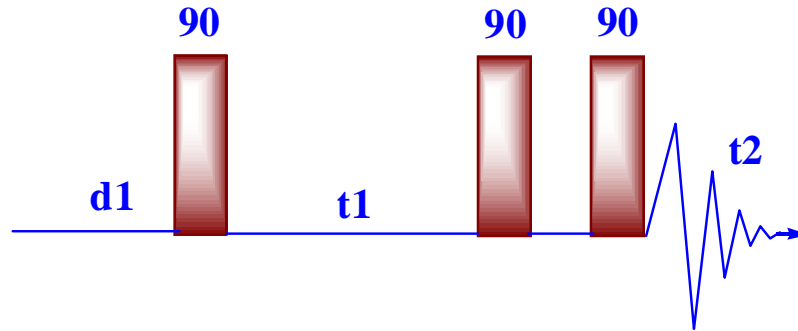


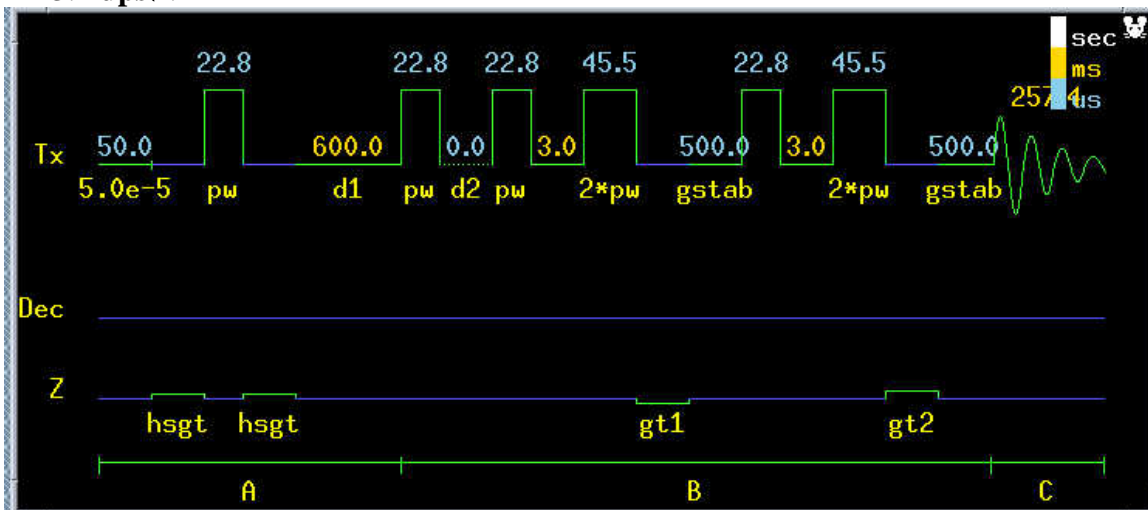
## Double quantum filtered COSY (DQF-COSY)



1. DQCOSY removes singlets therefore it is important if the solvent peaks are strong.
2. diagonal in pure adsorption mode with crosspeaks (cleans diagonal for close-in crosspeaks).
3. direct coupling is anti-phase, whereas indirect coupling is in-phase, allowing for  $J$  assignments in complex molecules.
4. Double quantum filtered COSY is phase-sensitive 2D NMR.
5. This pulse sequence converts the dispersive diagonal peak into antiphase absorption (like the cross peaks). A minimum of 8 scans is required for the phase cycling but if gradients are used to select the coherence pathway, only one scan is needed.

### Steps to do DQCOSY experiment.

1. run a standard 1D proton NMR. Calibrate **pw90** and move spectral width. Array pw and set **pw=pw90**.
2. **gdqcosy**.
3. **dps**.



4. **dg**.

ACQUISITION		TRANSMITTER		FLAGS		PROCESSING	
seqfil	gDQCOSY	tn	H1	hs	nn	sb	-0.257
sw	3978.7	sfrq	399.729	sspul	y	sbs	-0.256
at	0.257	tof	-397.9	hsglv1	4854	fn	2048
np	2048	tpwr	57	hsgt	0.002000	2D PROCESSING	
ss	32	pw	22.750	SAMPLE		sb1	-0.092
d1	0.600	DECOUPLER		date	Feb 25 2004	sbs1	-0.092
nt	1	dn	H1	solvent	CDC13	proc1	1p
ct	0	dm	nnn	sample	undefined	fn1	1024
2D ACQUISITION		GRADIENTS		SPECIAL			
sw1	3978.7	gz1v11	3640.5	temp	not used		
ni	428	gt1	0.002500	spin	not used		
phase	arrayed	gz1v12	7281	gain	26		
		gt2	0.002500				
		gstab	0.000500				

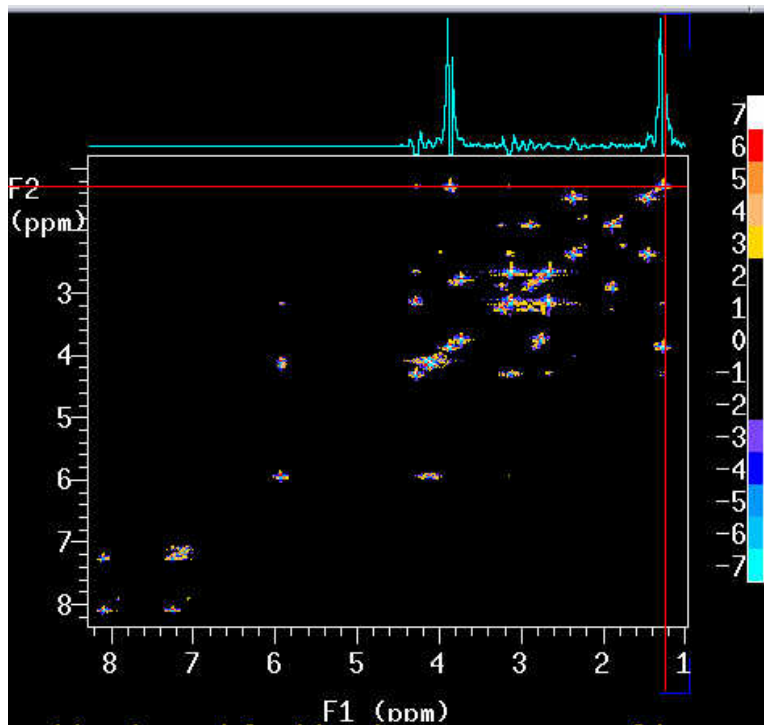
- Set **d1** = **0.6** (In this experiment, T1 for Strychnine is 200 ms, so 3T1 is 600 ms) (artifacts will result if **d1** is too small).
- sw1=sw**.
- ni** = **sw1/6 ~ sw1/12 (663~331)**. This is number experiments, or number of points in *t1*. The default value set by macro should be ok. If time allow, **ni** can be set to 600. This will increase the digital resolution on F1.  $dres1 \leq 6 \text{ Hz/pt} = sw1/(2ni)$ .
- np** = **2048**. This is number of points in *t2*. It costs nothing but disk space. It may increase at. But it is not a big deal. A big value gives better resolution in F2.
- fn=np** **fn1=2ni**. **fn** and **fn1** should be to be power of 2. So **fn1=1024**. Normally set none-zero-filling on F2 and one-zero-filling on F1.
- It is ok that **fn≠fn1** in **dqCOSY**. But but to baseline correct in both dimensions set **fn1=fn**. Also in data processing, foldt can only work when **sw1=sw** **fn=fn1**.
- nt** = **2** or **4**.
- go**.

### Phase-Sensitive 2d Data Workup and Plotting

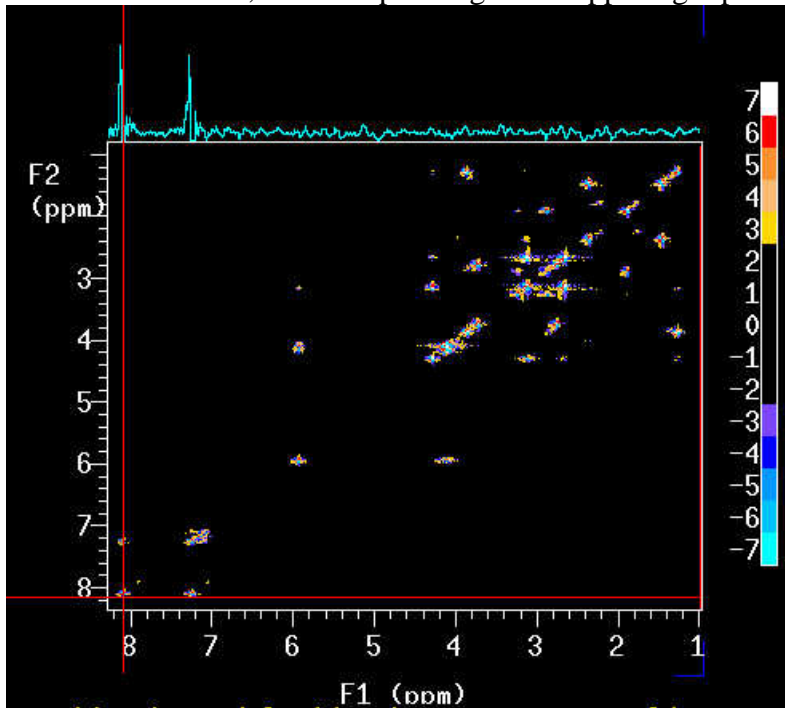
Often should not need to phase DQCOSY data at all.

- pmode='full'** This allows phasing along F2 in 2d spectrum.
- wft(1)** to display the first spectrum on *t1*.
- wtia** to open weighting function window. Adjust **lb**.
- wft1da** perform first transform (on *t2* dimension). **trace='f1'** **dconi**.
- click on **TRACE** and select strong intensity trace.
- wtia** to open weighting function window.
- adjust **lb/gf**.
- wft2da** performs 2D fourier transform.
- Place the mouse on the upper right peak **ds**.
- Phase this spectrum( only adjust the baseline)
- dconi**.
- Place the mouse button on the very left lower peak. **ds**.

13. Phase this spectrum. Please click on the right with no adjustment. This will keep the right phase (zero order) value and then move the left and phase the peak (first order).

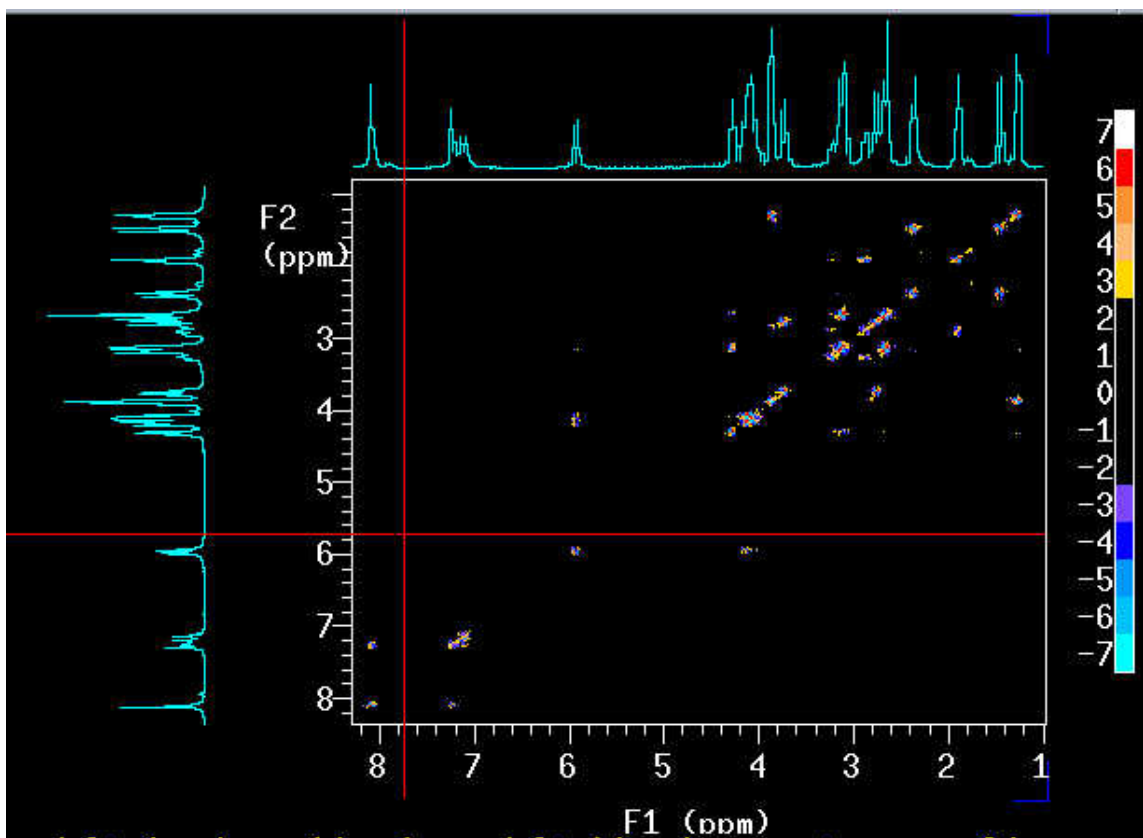


Look at the mouse, which is pointing at the upper right peak.



Mouse is pointing at the left-lower peak.

14. **dconi** ↵. This will display the final spectrum after phasing. You may type **trace='f2'** ↵ and phase the spectrum on F2.



To plot 2D spectrum with 1D on both sides as shown above, please do as follows:

1. In the 2D experiment, **dg1**↵. You will see the plotting information such as wc, sc, wc2, sc2.
2. **wc=140 wc2=140 dconi**↵.
3. **jexp1**↵. Where you did the 1D NMR with the same sw in the 2D experiment.
4. Display the 1D spectrum. Click **Main Menu**→**File**→choose the directory where you store the fid(s)→**set directory**. Click **Main Menu**→**File**→ choose the 1D fid→**load**→type **wft**↵.
5. **dg1**↵.
6. **wc=140. vp=148**↵. Adjust the vertical scale to avoid cutoff of peaks.
7. **pl**↵. This will plot 1D on top.
8. **wc2? wc2=140 vp=0 pl('side')** ↵. This will plot 1D on the side.
9. **jexp(2D)** ↵.
10. **pcon(10,1.2) page**↵.