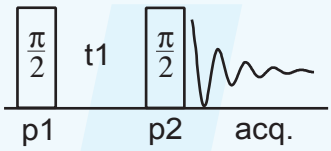
1. Introduction

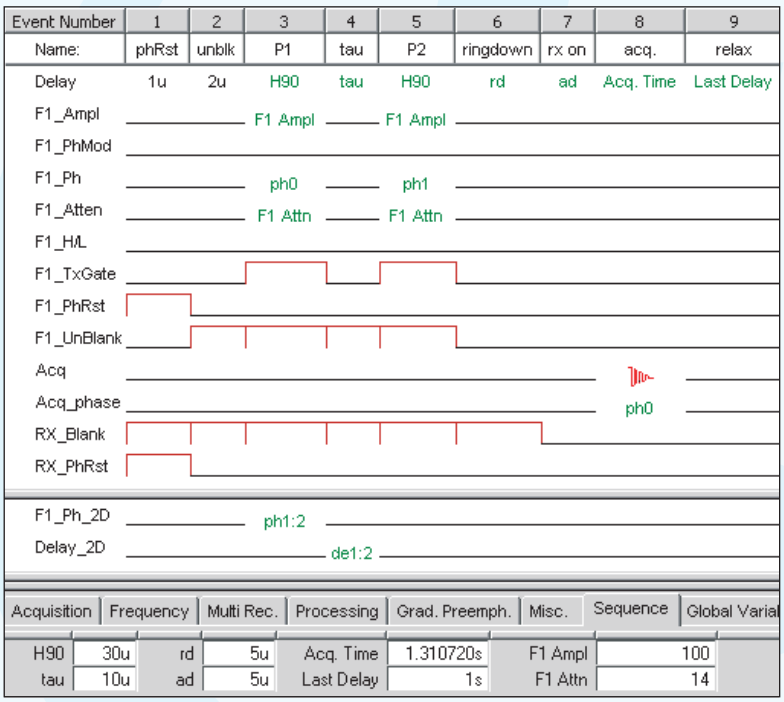
The COSY (COrrelation SpectroscopY) experiment generates 2D NMR correlation spectra. In the COSY sequence (Fig.1a), the first 90° pulse generates transverse magnetization. The second 90° pulse causes polarization transfer between J-coupled nuclei. For a two spin system with nuclei A and K, two peaks along the diagonal, (ω_A, ω_A) and (ω_K, ω_K), are observed if the spins are not J-coupled. If they are J-coupled, two additional cross-peaks appear at (ω_A, ω_K) and (ω_K, ω_A). Since coupled protons are typically separated by two or three covalent bonds, the chemical structure can be derived from COSY spectra. Described here is a phase sensitive COSY experiment using the States (also called the States-Haberkorn-Ruben, RuSH, or Hypercomplex) method. With this example, we demonstrate the basic procedure of 2D States phase sensitive NMR data acquisition and processing on Tecmag spectrometers.

2. Pulse sequence

(a)



(b)



1D phase tables:

ph0 (P1): 0, 0, 2, 2, 1, 1, 3, 3.

ph1 (P2): 0, 2, 1, 3, 2, 0, 3, 1.

2D phase tables:

ph1:2 (P1): 0, 1.

(All entries are in 4 step mode.)

2D delay table:

de1:2 (t1): Auto,

Use Dwell Time,

☐ Every 2 Passes.

(c)

scan	P1	P2	Receiver	Store as	
1	x	x	x	real	store as record 1
2	x	-x	x	real	
3	y	y	y	real	
4	y	-y	y	real	
5	-x	-x	-x	real	
6	-x	x	-x	real	
7	-y	-y	-y	real	
8	-y	y	-y	real	
9	y	x	x	imaginary	store as record 2
10	y	-x	x	imaginary	
11	-x	y	y	imaginary	
12	-x	-y	y	imaginary	
13	-y	-x	-x	imaginary	
14	-y	x	-x	imaginary	
15	x	-y	-y	imaginary	
16	x	y	-y	imaginary	

Fig. 1. (a) The pulse sequence for the COSY experiment. (b) The sequence in the NTNMR sequence editor with phase tables set for States method. The 2D phase table "ph1:2" is the phase cycle for P1 to generate real and imaginary records consecutively. The phase of P1 is the sum of the corresponding entries in tables, "ph0" and "ph1:2". The 2D delay table, "del:2", is used for t1 increments. The t1 values are 1dw, 1dw, 2dw, 2dw, ... , ndw, ndw in the 2D records, where dw is the dwell time and n is a half of Points 2D. (c) The phases for the first 16 scans for the complete phase cycling when Scans 1D is equal to 8.

3. Experiment and results

Sample:	2,3-dibromopropionic acid in benzene-D_6
Spectrometer:	7 Tesla magnet with Tecmag HF3 Discovery
Probe:	Nalorac D300-5 OWB 5mm $^{1}H/^{13}C$ Switchable probe
90° pulse:	30 µs
SW +/- (1D) :	390.6 Hz
SW 2D:	390.6 Hz
Dwell time (1D):	1.28 ms
Dwell_2D:	640 µs
Acq. points (and Points 1D):	1024
Points 2D:	1024
Scans 1D:	2

Notes:

The sample is not spun. The magnet is shimmed to ~0.5 Hz line width. If the magnet drift is greater than 0.5 Hz during the experiment, the sample should be locked. Set Dwell_2D = Dwell Time for the States Method. For best results, use at least 2 scans in Scans 1D.

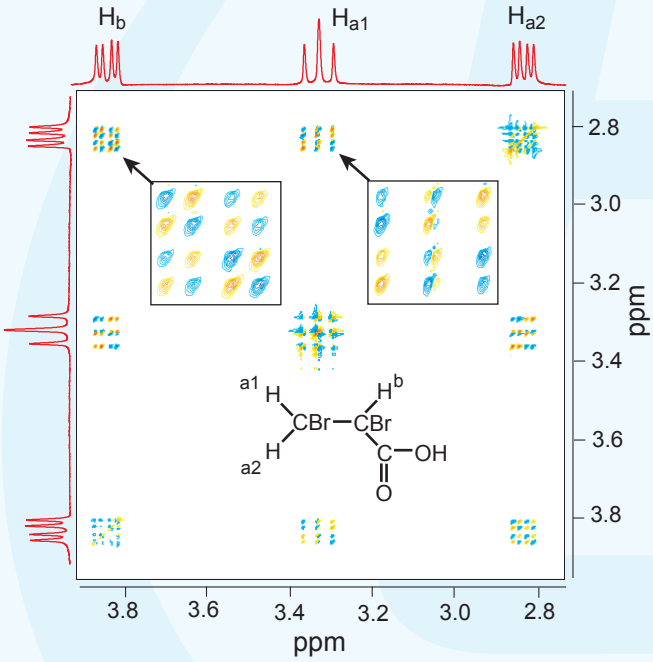


Fig. 2 The phase-sensitive COSY spectrum of 2, 3-dibromoproionic acid shown in benzene-D_6 in the regions of H_a and H_b. The 2D spectrum is obtained using the sequence in Fig. 1. The 1D spectrum is acquired using a single 90° pulse sequence and is shown on the top and left side.

Data processing:

1. Process the 1D data set (The System Phase will be used for 2D processing).
2. Process the first (t2) dimension of the 2D data set by opening the NDFT window and select: □□"Use 1D settings", "Fourier Transform", "Use System Phase", "Gaussian GB (1 Hz)", and click "Do it".□
3. Process the second (t1) dimension:
   a: to transpose the second into the first dimension, click Commands|Data Manipulation|Transform|Hyper-Complex;
   b. to remove every other records (the imaginary part in the original t2 dimemsion), click
   □□ i. Commands|Data Manipulation|Seperate Record|Joint (2),
   □□ ii. Commands|Data Manipulation|Read First Half;
   c. open the NDFT window and select "Use 1D settings", "Fourier Transform", "Gaussian GB (1 Hz)", and click "Do it",
   d. to transpose t1 back to the second dimension, □□ click Commands|Data Manipulation| Transform|Complex.
4. To display the processed data, click the 2D Display button and choose real data and contour plot.

The above procedure is automated in an NMRScript, "2D RuSH Processing". To use the script, first process the 1D spectrum, then click Scrits|Processing scripts| 2D RuSH Processing.

4. References

1. Derome, A. E, "Modern NMR Techniques for Chemistry Research", Pergamon Press, New York, 1987, p. 197-227.
2. D. J. States, R. A. Haberkorn, D. J. Ruben, *J. Magn. Reson*. **1982**, 48, 286-292.

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