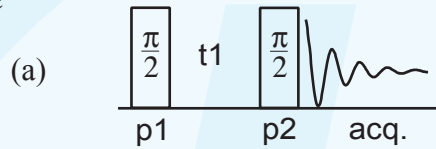


1. Introduction

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, ( $\omega_A, \omega_A$ ) and ( $\omega_K, \omega_K$ ), are observed if the spins are not J-coupled. If they are J-coupled, two additional cross-peaks appear at ( $\omega_A, \omega_K$ ) and ( $\omega_K, \omega_A$ ). 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



(b)

Event Number	1	2	3	4	5	6	7	8	9
Name:	phRst	unblk	P1	tau	P2	ringdown	rx on	acq.	relax
Delay	1u	2u	H90	tau	H90	rd	ad	Acq. Time	Last Delay
F1_Ampl			F1 Ampl		F1 Ampl				
F1_PhMod									
F1_Ph			ph0		ph1				
F1_Attn			F1 Attn		F1 Attn				
F1_HL									
F1_TxGate									
F1_PhRst									
F1_UnBlank									
Acq									
Acq_phase								ph0	
RX_Blank									
RX_PhRst									
F1_Ph_2D			ph1:2						
Delay_2D				del:2					
Acquisition	Frequency	Multi Rec.	Processing	Grad. Preemph.	Misc.	Sequence	Global Variat		
H90	30u	rd	5u	Acq. Time	1.310720s	F1 Ampl	100		
tau	10u	ad	5u	Last Delay	1s	F1 Attn	14		

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:

del:2 (t1): Auto,  
 Use Dwell Time,  
 Every 2 Passes.

(c)

scan	P1	P2	Receiver	Store as
1	x	x	x	real
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
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

store as record 1 (scans 1-8)  
 store as record 2 (scans 9-16)

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 <sub>6</sub>
Spectrometer:	7 Tesla magnet with Tecmag HF3 Discovery
Probe:	Nalorac D300-5 OWB 5mm <sup>1</sup> H/ <sup>13</sup> 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.

### Data processing:

- Process the 1D data set (The System Phase will be used for 2D processing).
- Process the first (t<sub>2</sub>) 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".
- Process the second (t<sub>1</sub>) dimension:
  - to transpose the second into the first dimension, click Commands|Data Manipulation|Transform|Hyper-Complex;
  - to remove every other records (the imaginary part in the original t<sub>2</sub> dimension), click
    - i. Commands|Data Manipulation|Separate Record|Joint (2),
    - ii. Commands|Data Manipulation|Read First Half;
  - open the NDFT window and select "Use 1D settings", "Fourier Transform", "Gaussian GB (1 Hz)", and click "Do it",
  - to transpose t<sub>1</sub> back to the second dimension,
    - click Commands|Data Manipulation|Transform|Complex.
- To display the processed data, click the 2D Display button and choose real data and contour plot.

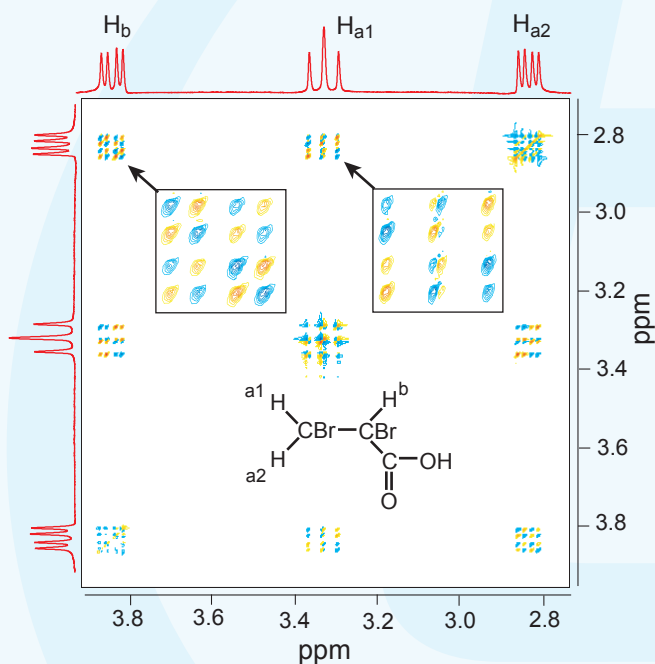


Fig. 2 The phase-sensitive COSY spectrum of 2, 3-dibromopropionic acid shown in benzene-D<sub>6</sub> in the regions of H<sub>a</sub> and H<sub>b</sub>. The 2D spectrum is obtained using the sequence in Fig. 1. The 1D spectrum is acquired using a single 90° pulse sequence and is shown on the top and left side.

## 4. References

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