BRUKER BIOSPIN
KOREA USER MANUAL

2D NMR MEASUREMENT

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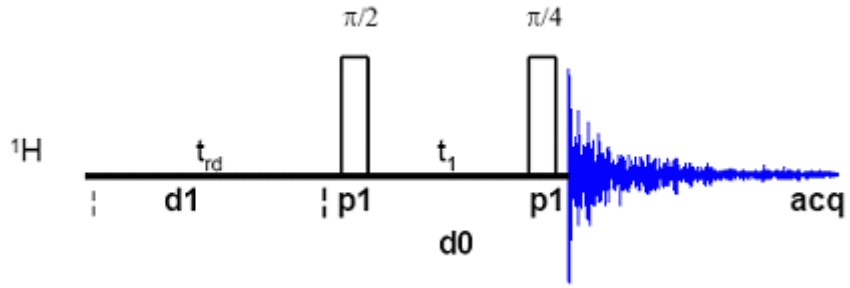
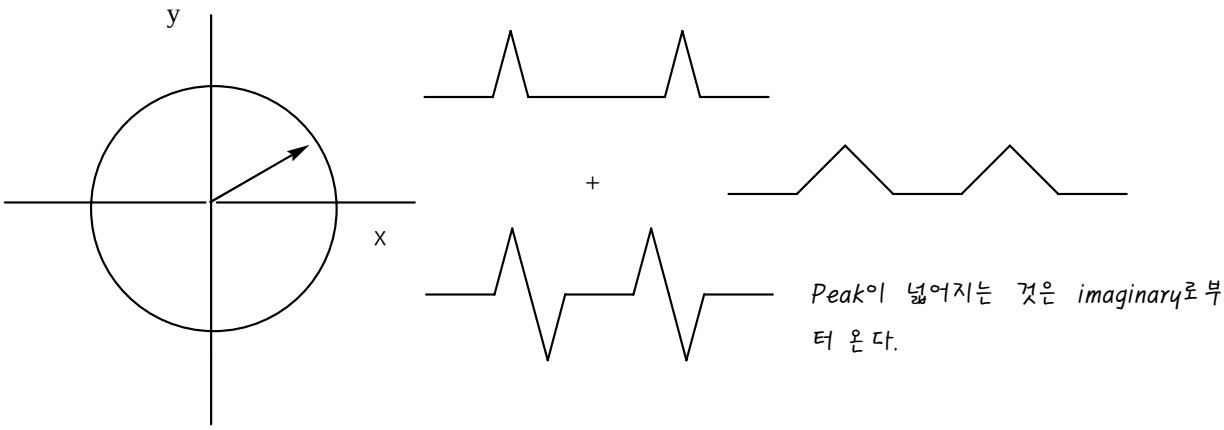
◎ Magnitude COSY



< COSY-45 Pulse Sequence>

- Principle

Pulprog : cosyqpgf (gp: gradient , gf: magnitude mode)



Magnitude → $\sqrt{x^2 + y^2}$

2D에서

◎ Record 1D Spectrum (Optimize O1 & SW)

- Spectrometer : F1 (1H)

- Preparation

1. **edc** (cosy/1/1)
2. Sample 넣기 : lift on,off
3. locking : **lock** → 용매선택
4. shim 값 부르기 : **rsh**
5. shim 조정 : Z , Z^2
6. Tuning , Matching : **wobb**
7. **getprosol**

- Set up acquisition parameter : eda

Parameter	Value	Comments
Pulprog	zg	
TD	32k	standard value
NS	1	
DS	0	
D1	2	default value
SW	20	미지시료일 경우
O1P	10ppm	

- Set up processing parameter : edp

Parameter	Value	Comments

SI	16 k	
LB	0.3 Hz	
PSCAL	global	

- Acquisition + Processing

- rga (receiver gain auto)
- zg (zero + go)
  - efp (em + ft + phase)
  - apk (auto phase correction)
  - abs (auto baseline correction)
  - sref (set reference)
  - phase 맞춤 : ph01 , ph02
  - 관심있는 영역 확대하여 dp1 : F1 , F2 , PSCAL=yes , sw-sf01
  - plotreg

◎ Record COSY Spectrum

- Preparation

1. iexpno (increment experiment number , cosy/2/1)
2. parmode ⇒ 2D 선택

- Set up acquisition : eda or ased

Parameter	F2	F1	Comments
Pulprog	cosyqf		
TD	2K	256	
NUC1		1H	
ND0		1	# of delay d0
Phase mode		Fnmode = QF	not phase sensitive
NS	8		4×n
DS	16		
p1(pl1)			90 pulse length & power in F1 channel
D0	3		
D1	3		default value
SW	ID SW	F2 와 같은값	

- Acquisition

- rga (receiver gain auto)

- zg (zero + go)

※ expt: experiment time

- Set up processing : edp

Parameter	F2	F1	Comments
SI	>=TD(F2) , **2K**	=2×TD(F1) , **512**	

WDW	SINE	SINE	
SSB	0	0	# of delay d0
PH_mod	no	mc	magnitude calculation
PKNL	TRUE		디지털 필터 사용시 필요
BC_mod	quad	no	
MC2		QF	determine type of FT: QF results in a forward quadrature complex FT

- Processing

- xfb (2D-FT in F2+F1 - dimension)

※ xf2 (Ft in F2-dimension) , xf1 (FT in F1-dimension)

- abs2 (auto-baseline in F2-dimension)

	adjust the threshold level
	change the positive/negative peaks
	expand the interests region

- abs1 (auto-baseline in F1-dimension)

- defplot (최적화된 화면 저장)

Change levels? y

Please enter number of positive levels? 6

Display contours? n

- Plotting the Spectrum

- rpar standard2D plot (적합한 plot parameter 값 부르기)

- edg

- Click **ed** button of **EDPROJ1**

```
PF1DU       u
PF1USER     (name of user for file cosy/1/1)
PF1NAME     cosy
PF1EXP      1
PF1PROC     1
```

- Click **ed** button of **EDPROJ2**

```
PF2DU       u
PF2USER     (name of user for file cosy/1/1)
PF2NAME     cosy
PF2EXP      1
PF2PROC     1 .
```

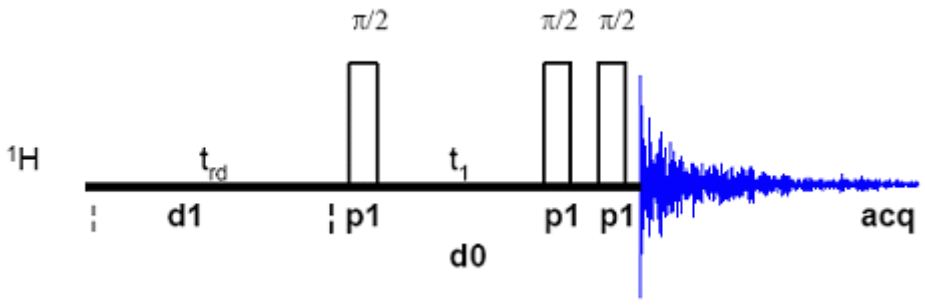
- Calibrate refrence peak is as 0ppm

- setti

- view or xwinplot

- plot

◎ Double-Quantum Filtered (DQF) COSY

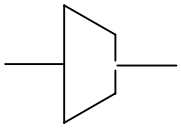


<DQF-COSY Pulse Sequence>

- Principle

- phase sensitive mode : good resolution (※ broad peak in magnitude mode)

- mutiple quantum coherence after second pulse

FID는  형태로 얻어진다. 여기에 WDW를 곱해서 일반적인 FID 모양을 같게 한다. 위의 형태를 그냥 ft하면 wiggling이 생긴다

cosy에서는 RG를 하지 않는다.
그림 생략
FID의 형태가 위와 같이 나오기 때문에 RGA를 하게되면 signal을 전부 잃어버릴 수 있다.

Rg 값을 얻는 방법
1D로부터 얻어온다.
gs에서 얻는다

DQF cosy는 큰 rg값을 가져야 한다(1k, 2k, 3k, 4k, ……)

◎ Record 1D Spectrum (Optimize O1 & SW)

◎ Record COSY Spectrum

- Preparation

- edc : cosy/3/1 , (from cosy/2/1)

- Set up acquisition : eda

Parameter	F2	F1	Comments
Pulprog	**cosydfph**		
TD	2K	512	
NUC1		1H	
ND0		1	# of delay d0

Phase mode		Fnmode = States-TPPI	not phase sensitive
NS	16		4×n
DS	16		
p1(pl1)			90 pulse length & power in F1 channel
D0	3		
D1	3s		default value
SW	ID SW	F2 와 같은값	

- Set up processing : edp

Parameter	F2	F1	Comments
SI	>=TD(F2) , **2K**	=2×TD(F1) , **1K**	
WDW	SINE	SINE	
SSB	2	2	# of delay d0
PH_mod	pk	pk	phase correction
PKNL	TRUE		디지털 필터 사용시 필요
BC_mod	no	no	
MC2		States-TPPI	States-TPPI results in a foward complex FT

- Acquisition + Processing

- gs (interactive go)

: 최대 fid 진폭 높이가 화면의 1/2 정도 되게 **rg** 값 조정

- zg (zero + go) ※ no rga

- Processing

- xfb (2D-FT in F2+F1 - dimension)

※ xf2 (Ft in F2-dimension) , xf1 (FT in F1-dimension)

- abs2 (auto-baseline in F2-dimension)

	adjust the threshold level
	change the positive/negative peaks
	expand the interests region

- abs1 (auto-baseline in F1-dimension)

- phase (1.row , 2.column)

※ cross peak 로 phase 맞춘다. (not digonal peak)

※ dqf-cosy 의 peak 는 anti-phase 이다. (positive + negative)

- defplot (최적화된 화면 저장)

Change levels? y

Please enter number of positive levels? 6

Display contours? n

- Plotting the Spectrum

- rpar standard2D plot (적합한 plot parameter 값 부르기)

- edg

- Click **ed** button of **EDPROJ1**

PF1DU	u
PF1USER	(name of user for file cosy/1/1)
PF1NAME	cosy
PF1EXP	1
PF1PROC	1

- Click **ed** button of **EDPROJ2**

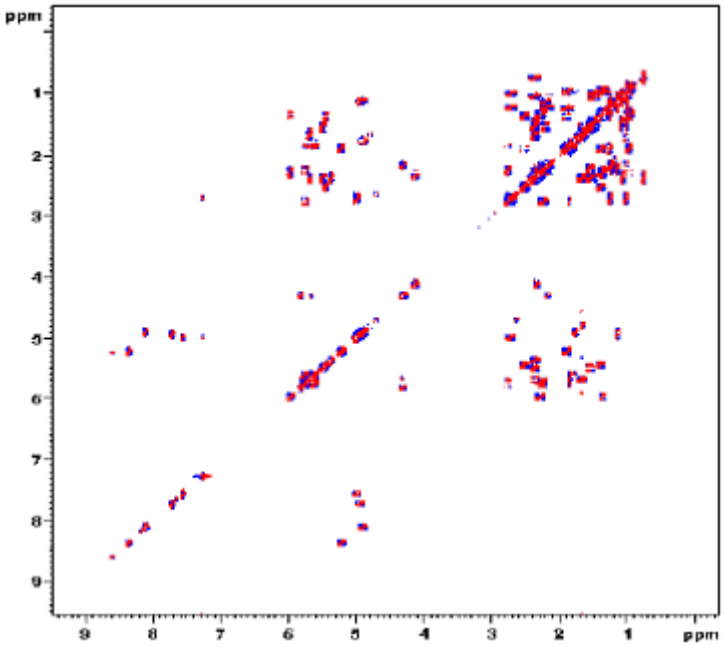
PF2DU	u
PF2USER	(name of user for file cosy/1/1)
PF2NAME	cosy
PF2EXP	1
PF2PROC	1 .

- Calibrate refrence peak is as 0ppm

- setti

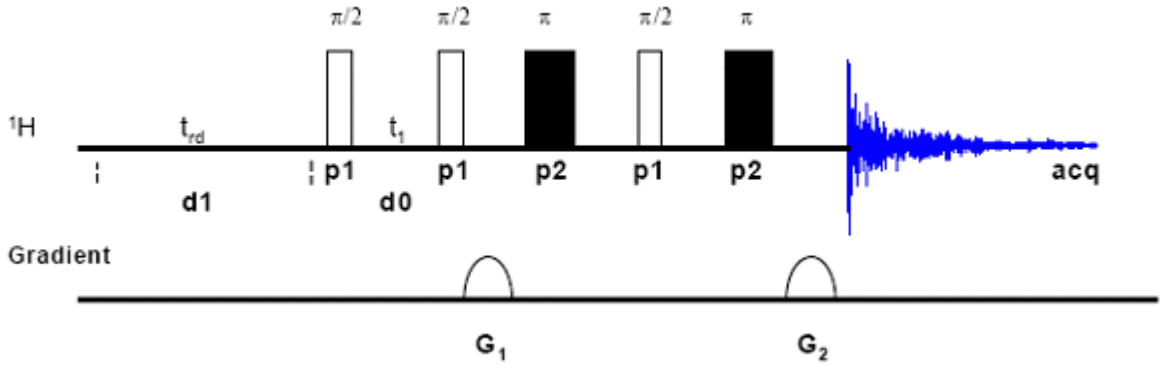
- view or xwinplot

- plot



DQF-COSY Spectrum of 50 mM Cyclosporin in C6D6

◎ DQF-COSY using Pulsed Filed Gradient : GRASP-DQF-COSY



GRASP-DQF-COSY Pulse Sequence

- Principle

- phase cycling 대신 gradient pulse 사용 (more efficient)

- NS ↓, 시간 ↓ (more efficient than phase cycling)

- quadrature detection in F1 dimension

- double quantum filter: solvent peak (H2O) can be suppressed efficiently without any other solvent suppression technique

- 두 번째 90°: multiple quantum coherences

- 첫 번째 G1: complete dephasing of all coherence

- 세 번째 90°: convert multiple-quantum into observable single quantum coherence

- 두 번째 G2: rephase all observable peaks

※ G1:G2=2:1 ⇒ double quantum filter , G1:G2=3:1 ⇒ triple quantum filter

- Preparation

- record 1D spectrum

  - **edc** : (from 1D)

- Set up acquisition : eda

Parameter	F2	F1	Comments
Pulprog	**cosygpmfph**		
TD	2K	512	
NUC1		1H	
ND0		1	# of delay d0
Phase mode		Fnmode = TPPI	
NS	4		4×n
DS	16		
p1(pl1)			90 pulse length & power in F1 channel
p16	1.5m		Length of gradient pulse
D0	3		
D1	3s		default value
D16	150u		Gradient recovery delay
gpz1	10		% of the maximum gradient amplitude
gpz2	20		
gpname1	SINE.100		Gradient shape
gpname2	SINE.100		〃
SW	ID SW	F2 와 같은값	

- Set up processing : edp

Parameter	F2	F1	Comments
SI	>=TD(F2) , **2K**	=2×TD(F1) , **1K**	
WDW	SINE	SINE	
SSB	2	2	# of delay d0
PH_mod	pk	pk	phase correction
PKNL	TRUE		디지털 필터 사용시 필요
BC_mod	no	no	
MC2		TPPI	States-TPPI results in a foward complex FT

- Acquisition + Processing

- gs (interactive go)

: 최대 fid 진폭 높이가 화면의 1/2 정도 되게 **rg** 값 조정

- zg (zero + go) ※ no rga

- Processing

- xfb (2D-FT in F2+F1 - dimension)

※ xf2 (Ft in F2-dimension) , xf1 (FT in F1-dimension)

- abs2 (auto-baseline in F2-dimension)

	adjust the threshold level

+/-	change the positive/negative peaks
	expand the interests region

- abs1 (auto-baseline in F1-dimension)

- phase (1.row , 2.column)

※ cross peak 로 phase 맞춘다. (not digonal peak)

※ dqf-cosy 의 peak 는 anti-phase 이다. (positive + negative)

- defplot (최적화된 화면 저장)

Change levels? y

Please enter number of positive levels? 6

Display contours? n

- Plotting the Spectrum

- rpar standard2D plot (적합한 plot parameter 값 부르기)

- edg

- Click **ed** button of **EDPROJ1**

```
PF1DU       u
PF1USER     (name of user for file cosy/1/1)
PF1NAME     cosy
PF1EXP      1
PF1PROC     1
```

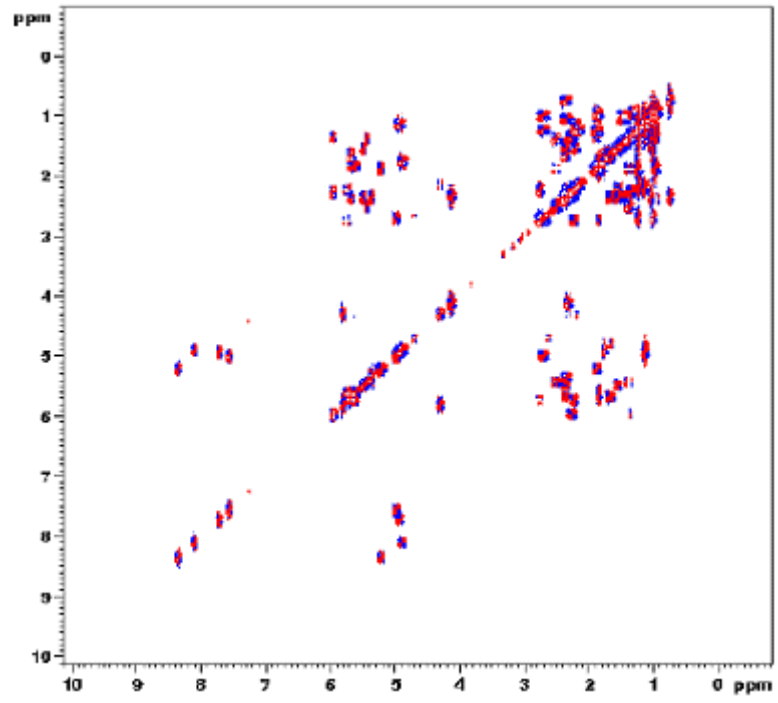
- Click **ed** button of **EDPROJ2**

PF2DU u
PF2USER (name of user for file cosy/1/1)
PF2NAME cosy
PF2EXP 1
PF2PROC 1 .

- Calibrate refrence peak is as 0ppm

- setti

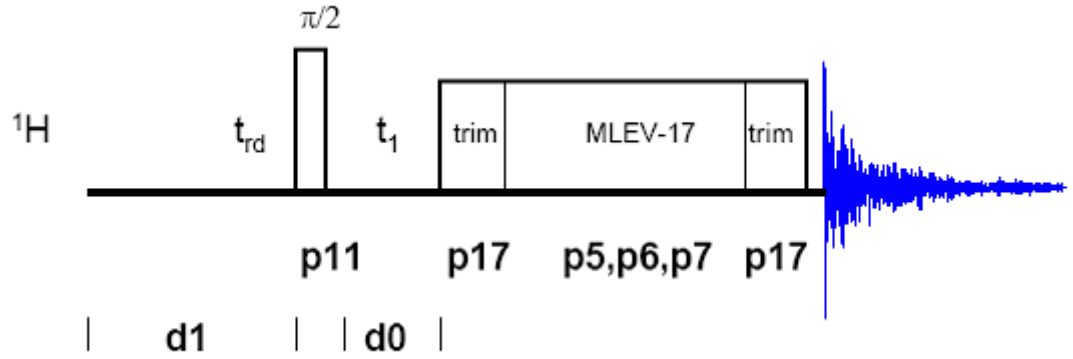
- view or xwinplot



- plot

GRASP-DQF-COSY experiment of 50mM Cyclosporin in C6D6

◎ TOCSY (TOtal Correlation SpectroscopY)



TOCSY Pulse Sequence

- Principle

- phase cycling 대신 gradient pulse 사용 (more efficient)

- NS ↓, 시간 ↓ (more efficient than phase cycling)

- quadrature detection in F1 dimension

- double quantum filter: solvent peak (H2O) can be suppressed efficiently without any other solvent suppression technique

- 두 번째 90°: multiple quantum coherences

- 첫 번째 G1: complete dephasing of all coherence

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- 두 번째 G2: rephase all observable peaks

※ G1:G2=2:1 ⇒ double quantum filter , G1:G2=3:1 ⇒ triple quantum filter

- Preparation

- record 1D spectrum

- **edc** : (from 1D)

- Set up acquisition : eda

Parameter	F2	F1	Comments
Pulprog	**cosygpmfph**		
TD	2K	512	
NUC1		1H	
ND0		1	# of delay d0
Phase mode		Fnmode = TPPI	
NS	4		4×n
DS	16		
p1(pl1)			90 pulse length & power in F1 channel
p16	1.5m		Length of gradient pulse
D0	3		
D1	3s		default value
D16	150u		Gradient recovery delay
gpz1	10		% of the maximum gradient amplitude
gpz2	20		
gpname1	SINE.100		Gradient shape
gpname2	SINE.100		〃

SW	ID SW	F2 와 같은값	

- Set up processing : edp

Parameter	F2	F1	Comments
SI	>=TD(F2) , **2K**	=2×TD(F1) , **1K**	
WDW	SINE	SINE	
SSB	2	2	# of delay d0
PH_mod	pk	pk	phase correction
PKNL	TRUE		디지털 필터 사용시 필요
BC_mod	no	no	
MC2		TPPI	States-TPPI results in a foward complex FT

- Acquisition + Processing

- gs (interactive go)

: 최대 fid 진폭 높이가 화면의 1/2 정도 되게 **rg** 값 조정

- zg (zero + go) ※ no rga

- Processing

- xfb (2D-FT in F2+F1 - dimension)

※ xf2 (Ft in F2-dimension) , xf1 (FT in F1-dimension)

- abs2 (auto-baseline in F2-dimension)

	adjust the threshold level
	change the positive/negative peaks
	expand the interests region

- abs1 (auto-baseline in F1-dimension)

- phase (1.row , 2.column)

※ cross peak 로 phase 맞춘다. (not digonal peak)

※ dqf-cosy 의 peak 는 anti-phase 이다. (positive + negative)

- defplot (최적화된 화면 저장)

Change levels? y

Please enter number of positive levels? 6

Display contours? n

- Plotting the Spectrum

- rpar standard2D plot (적합한 plot parameter 값 부르기)

- edg

- Click **ed** button of **EDPROJ1**

```
PF1DU      u
PF1USER    (name of user for file cosy/1/1)
PF1NAME    cosy
PF1EXP     1
PF1PROC    1
```

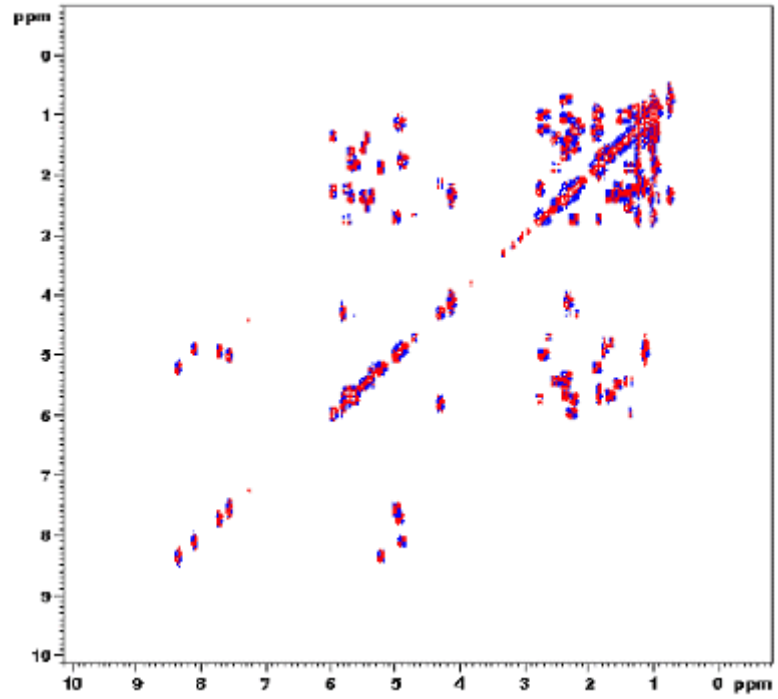
- Click **ed** button of **EDPROJ2**

PF2DU u
PF2USER (name of user for file cosy/1/1)
PF2NAME cosy
PF2EXP 1
PF2PROC 1 .

- Calibrate refrence peak is as 0ppm

- setti

- view or xwinplot



- plot

GRASP-DQF-COSY experiment of 50mM Cyclosporin in C6D6